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A Novel Approach to Mitigating the Potential Release of Radioisotopes Under Fire Conditions - Enhancing Fire Resiliency of Radiological Contamination Fixatives During Deactivation & Decommissioning Activities

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EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL), in close collaboration with the Florida International University Applied Research Center (FIU ARC), successfully executed a technology development activity on behalf of the Department of Energy, Office of Environmental Management (DOE EM). The purpose of the activity was to improve the capability of fixative technologies in immobilizing residual contamination when exposed to thermal stressors as postulated in accident scenarios in Basis for Interim Operations (BIO) documents across the DOE EM complex. The effort resulted in the test and evaluation of a down-selected, commercial-off-the-shelf (COTS) intumescent technology in a radioactive environment at the Savannah River Site (SRS) Building 235-F Plutonium Fuel Form (PuFF) Facility and highlighted the potential use of this technology as a stand-alone, fire retardant fixative. Furthermore, this activity highlighted significant shortfalls in common fixatives currently used to support decommissioning activities and brought to the forefront the need for a methodical, uniformed approach to certifying fixative technologies for operational use in Deactivation and Decommissioning (D&D) activities.

An essential component of this research was to evaluate the state of industry fixatives currently in use and set the foundation for comparison to alternative technological solutions. A baseline of five (5) commonly used fixatives and decontamination gels (hereafter collectively referred to as fixatives) was conducted, and notable shortfalls and deficiencies were revealed, particularly when exposed to thermal, water, and other environmental stressors. At temperatures as low as 300 °F - 400 °F (148.89 °C – 204.44 °C), all commonly used fixatives melted from the substrates within 3-5 minutes of exposure, resulting in contaminant transport. Significant degradation in terms of mass loss, desiccation, and off-gassing occurred, and the chemical breakdown of the polymer was so complete researchers assessed there would likely have been a release of residual contamination. Additional vulnerabilities became evident when exposed to water immersion and high humidity; existing fixatives took up water, swelling and frequently delaminating from the substrate, once again increasing the likelihood of a contaminant release.

Researchers leveraged their firsthand experience in the use of intumescent coating technologies to improve fire protection of facilities in support of the U.S. military, and hypothesized that intumescent materials could be adapted to enhance fire resiliency in fixatives and potentially mitigate the release of radioisotopes when exposed to fire and/or extreme heat. Based on a series of successful proofs-of-concept, a specific intumescent technology was down-selected due to its easy application and potential as a standalone fixative. Extensive coordination with SRS 235-F site personnel resulted in a hot demonstration of the material in a designated Pu-238 contaminated process cell and hood. The demonstration proved the material could be applied in a radiological environment with appropriate containment and that the material can hold up for a 2 year period provided it is allowed to cure without objects or absorbent spills on top of the freshly applied layers. Lessons learned were identified and, when incorporated in future applications, it is assessed the technology will provide a marked improvement in operational performance, particularly when compared to current fixatives being used by sites for D&D. Future work was also identified as necessary to quantify the materials behavior to stressors outlined in the DOE-HDBK-3010 including seismic/vibration, impact, etc. to provide source data for Complex-level D&D guidance updates.

Perhaps the most significant finding in the execution of this activity was the lack of a systemic, uniform methodology for certifying and crediting fixative technologies. The operational requirement for standards-based performance specifications and testing protocols was consistently emphasized throughout the test and evaluation process by safety basis personnel and end users. Adherence to international standards significantly reduces risk for the end-user through uniformity, standardization, and quality assurance in technology tests and evaluations. However, uniform testing protocols for critical aspects of evaluating fixative technologies were not available, and continued engagement with international standards organizations is required. It was also found that use of similar or "in kind" standards resulted in technical compliance of standard certification; however, these standards were found to be insufficient in application as contamination fixatives. Secondly, the guiding document for determining the Source Term (DOE-HDBK-3010), has not been updated for nearly 30 years, and fails to account for the positive impacts that certified fixative technologies have on reducing airborne release fractions (ARFs) and respirable fractions (RFs). Outdated assumptions used in calculating the source term formula do not allow end users to take appropriate credit for lowering these coefficients and serve as a disincentive for deploying the technology during D&D activities.

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LIST OF ABBREVIATIONS

ABL	Actinide Billet Line
ACM	Asbestos-Containing Material
ARC	Applied Research Center
ARF	Airborne Release Fraction
ASTM	American Society of Testing and Materials
BIO	Basis for Interim Operations
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COTS	Commercial-off-the-shelf
CPS	Centipoise
D&D	Deactivation and Decommissioning
DI	Deionized
DNFSB	Defense Nuclear Facility Safety Board
DOE	Department of Energy
EM	Office of Environmental Management
EPA	Environmental Protection Agency
FIU	Florida International University
FM	Factory Mutual
GAO	Government Accountability Office
GC-MS	Gas Chromatograph-Mass Spectrometer
HEPA	High Efficiency Particulate Air
IC	Intumescent Coating
ISO	International Organization for Standardization
MAR	Material at Risk
MSL	Mean Sea Level
NFPA	National Fire Protection Association
OML	Old Metallurgical Laboratory
OSHA	Occupational Safety and Health Administration
PEF	Plutonium Experimental Facility
PuFF	Plutonium Fuel Form
R&D	Research and Development
RCA	Radiologically Controlled Areas
RF	Respirable Fractions
RH	Relative Humidity
S&M	Surveillance and Maintenance

SCDHEC	South Carolina Department of Health and Environmental Control
SDS	Safety Data Sheet
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
THF	Tetrahydrofuran
UBC	Uniform Building Code
UL	Underwriter Laboratories
ULC	Underwriters Laboratories of Canada
UV	Ultraviolet
VOC	Volatile Organic Compound
VSDS	Visual Survey Data System
WAC	Waste Acceptance Criteria

1.0 Introduction

Nuclear facilities that are moving towards final disposition face enormous challenges to ensure no environmental release of holdup material occurs between the time the facility is no longer active and final disposition is achieved. Workers actively seek to remove as much of the radioactive holdup as possible; however, decontamination can only remove so much of the material before further efforts become time and/or cost prohibitive. As such, for contaminated areas, there is often residual contamination remaining after material removal and decontamination operations are completed.

To overcome free standing contamination, it is sometimes favorable to place a fixating layer to prevent residual contamination release. These fixating materials are often in the form of polymer layers labeled "fixatives" by the manufacturer. There is little to no data for these materials pertaining to their response to environmental conditions outside of manufacturer specified "ideal conditions" (~75 ºF (23.89 ºC), 40% relative humidity). For facilities undergoing deactivation and decommissioning (D&D) activities, however, these ideal conditions cannot be expected as facility upkeep and environmental stability systems will inevitably be reduced or taken offline. The lack of operational testing and evaluation of commonly-used fixatives, particularly when exposed or subjected to scenarios postulated in the Basis for Interim Operations (BIO) at facilities across the Department of Energy, Office of Environmental Management (DOE EM) Complex, was highlighted by DOE EM's Office of Infrastructure and D&D by then director Mr. Andrew Szilagyi to Savannah River National Laboratory (SRNL) and Florida International University (FIU) researchers. Assessing their performance in non-ideal environments often associated during D&D, such as high heat and humidity, as well as their performance during accident scenarios such as a seismically-induced full-facility fire and other natural-hazard events, emerged as an operational necessity in order to ascertain any potential vulnerabilities. This testing would, in turn, promote a better understanding of current fixative technologies and their performance in an array of conditions common to D&D, better inform the development of mitigation strategies for safety basis personnel who use fixatives, and potentially identify new technological solutions to improve the operational performance and confidence of fixatives in addressing postulated contingency scenarios in the facility's BIO.

2.0 Background

2.1 D & D Overview

After the end of their missions or operational lives, process contaminated facilities undergo "disposition", which is a series of stages leading to a final end state of demolition, in-situ disposal, or a combination of both. The D&D process aims to mitigate risks to the workers remaining in the area, the public, and the environment in support of facility disposition. Deactivation occurs after a facility has been transitioned from operations and entered a safe and stable shutdown condition. Deactivation focuses on achieving a low-risk and low-cost state for the facility which requires minimal surveillance and maintenance (S&M) until final disposition is complete. This portion of the facility transition begins by removing/disabling routine facility capabilities as applicable, such as electricity, environmental control, and ventilation where appropriate without risking radiological material release. Another part of the deactivation process includes reducing remaining hazards (both radiological and industrial) identified during facility shutdown. Deactivation activities such as removal of process material, stabilization, decontamination, and isolation of the facility and its systems are performed to bring the facility to a safe storage state. The goal of these activities is risk reduction to a collocated worker, the public, the environment, and to minimize required S&M activities which continue until a facility is fully decommissioned and reaches its final disposition. Final disposition could be decades from the end of a facility's mission or its operational shutdown, meaning these hazards exist in a non-functioning facility for many years in degraded and reduced infrastructure and non-ideal environmental conditions with limited surveillance. Decommissioning is typically the final stage of disposition. This is when any unacceptable residual hazards are eliminated permanently. The decommissioning stage should bring the facility itself to its final end state.[1]

2.1.1 Regulatory Requirements

There are significant regulatory requirements that must be met through the disposition period. For example, state and federal agencies such as the Department of Energy (DOE), the Environmental Protection Agency (EPA), and South Carolina Department of Health and Environmental Control (SCDHEC), are all regulatory bodies with oversight of D&D activities at the Savannah River Site (SRS), and any site performing D&D of a facility will be subject to similar regulatory bodies. Federal regulations impacting D&D at SRS primarily include 10 CFR Part 830: Nuclear Safety Management, 29 CFR 1926 Safety and Health Regulations for Construction, and the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). DOE standards such as DOE-STD-3009 Preparation of Nonreactor Nuclear Facility Documented Safety Analysis, DOE-STD-1120-2016 Preparation of Documented Safety Analysis for Decommissioning and Environmental Restoration Activities, and DOE handbooks and manuals like DOE-HDBK-3010-94, DOE/EM-0318 Facility Deactivation Guide Methods, and Practices Handbook further dictate requirements for D&D. Interpretation on how to meet the outline requirements is left to the site and contractors to develop and negotiate with relevant governing parties to ensure proper considerations and regulations have been met. At SRS, internal manuals and procedures like SRNS Facility Disposition Manual 1C, the implementing procedure used at SRS for DOE Orders 430.1C and DOE Guides 430.1-4, provide site level guidance for regulations impacting disposition activities.[1-9]

Pertaining to fixative use in the D&D process, there is no firm regulatory guidance allowing a facility or site to certify or credit the use of such material. Consequently, they are predominantly used as a “best practice,” but do not have to comply with any regulatory performance standards, leaving a potential risk of release for any improperly selected materials based on an assumed performance. As such, use of such materials may give a false sense of safety when utilized, as the specific performance metrics for the material in a given facility or area are not benchmarked, and may fail unexpectedly depending on the conditions they are subjected to. To allow a facility to credit fixative technologies in the D&D process, an intricate web of state, federal, and internal site requirements where D&D is occurring must be navigated while also considering Complex-wide concerns in these areas.

2.1.2 Gross Decontamination Approaches

Deactivation of a facility encompasses a series of actions that will collectively reduce the residual hazards in the facility and/or reduce the cost of custodial care of the facility (e.g., S&M, operation of essential equipment, etc.). Reducing material at risk (MAR) is often a significant concern in deactivation efforts, and radiological decontamination is therefore a key activity. MAR is material that could be released from a facility, impacting personnel or the environment, and includes both radiological and industrial hazards. For the purposes of this report, decontamination and subsequent fixative application is discussed in terms of radiological contaminants. Radiological decontamination during deactivation can take many forms, but typical approaches focus on mechanical, chemical, and cyclic energy methods.[10]

Mechanical methods make use of physical or mechanical abrasion of the contaminant or the contaminated surface. Mechanical decontamination methods and technologies include blasting, shaving, dry and steam vacuuming, scabbling, brushing/sweeping, and application and removal of strippable coatings.[11] Mechanical methods are often preferred, with simpler methods chosen over more complex due to the worker hazards or impacts to the facility/infrastructure associated with the methods. Chemical methods make use of chemical reagents or manipulation of chemical properties of the contaminant or contaminated surface for removal. Examples of chemical methods include the use of chelation and organic acids, strong mineral acids, chemical foams and gels, and oxidizing and reducing agents.[12] These methods generate a significant amount of liquid waste that must be subsequently stabilized, making these methods of decontamination less attractive than more rudimentary mechanical means, however they can provide significant decontamination of a surface in fewer applications/timeframes. Cyclic energy methods make use of incident energy in the form of sound, electrical, or radiation to separate contamination from contaminated surfaces.[11] This is known as electrochemical-based decontamination, and yield even greater decontamination, however the greater worker hazards and difficulty in targeted application in most facilities typically preclude use of these methods except in extremely favorable circumstances.

2.1.3 Technical and Non-technical Challenges with Residual Contamination and Fixatives

Nuclear facilities that are moving towards final disposition face enormous challenges to ensure no residual contamination or holdup material is released to the environment between the time the facility is no longer active until final disposition is achieved. Workers actively seek to remove as much of the radioactive holdup as possible; however, decontamination can only remove a fraction of the material before further efforts become time/cost prohibitive. As such, for contaminated areas, there is often residual contamination remaining after material removal and decontamination operations are completed.[13] This residual contamination still poses a hazard, and effectively immobilizing it until the facility reaches its final end state constitutes a significant technical challenge. Further complicating this effort is the requirement to fix this residual contamination under a variety of postulated accident scenarios in facility BIOs. Events such as seismic activity and fire, for example, pose remarkable threats to facilities during the storage phase of disposition and could lead to the release of radiological contamination.

Technological solutions, like the development of fixatives that can mitigate the spread and continue to immobilize residual contamination even when exposed to a host of stressors (i.e.: thermal, impact, environmental, and water immersion) can provide a cost effective solution to long-term stability of residual material. Several non-technical challenges associated with the use of fixative technologies also exist that have been neglected, but significantly impact their development, deployment, and adoption across the DOE EM Complex. Chief among these is a lack of standardized certification protocols for fixative materials used in D&D. As highlighted by past U.S. Government Accountability Office (GAO) reports, site officials may not be familiar with innovative technologies and fear their use would present an unacceptable risk or be unacceptable to regulators, or could result in missing milestones if the technology fails to perform as expected.[14] The lack of universally-accepted standard specifications, performance metrics and uniform testing protocols for fixative technologies make it exceedingly difficult to certify them against a known performance standard/metric. This shortfall ultimately increases the risk for end users because the material cannot be credited and is not well-defined, therefore any material that appears to fixate the residual contamination can be used by contractors as a “best practice.”

A second non-technical challenge discovered over the course of this work involves the aged DOE Handbook on Airborne Release Fractions/Rates and Respirable Fractions for Nonreactor Nuclear Facilities (DOE-HDBK-3010).[7] This directive, used extensively in calculating the Source Term of a facility, has not been updated since its initial publication in 1994, and therefore fails to account for and credit the positive, quantifiable effects fixative technologies have on reducing the airborne release fraction (ARF) and respirable fraction (RF) calculations required by DOE. Though the DOE-HDBK-3010 gives guidance on appropriate factors for dealing with the residual contamination that is often in powder form, there is no guidance on how to appropriately credit technologies such as fixative platforms that immobilize the contamination in a solid polymer material under normal operating conditions or when exposed to thermal and impact stressors. As such, overly conservative ARFs and RFs are applied that assume any powder, fixed or not, has free mobility, which in turn requires the implementation of challenging operational restrictions and costly nuclear safety controls. This has the additional effect of presenting significant obstacles to introducing new, state-of-the-art fixative technologies intended to support D&D activities since the DOE-HDBK-3010 does not account for their effects on reducing various risks in safety basis calculations per the Source Term Formula.

2.2 Decommissioning / Risk Reduction of 235-F

2.2.1 Description of Building

Building 235-F was constructed near the center of SRS in the 1950's as part of the original Savannah River Plant and has been used for a variety of missions since that time. The original mission for the building was to act as the C-line which would take Pu-239 metal and produce triggers for nuclear weapons, but this mission was canceled before any equipment was installed and the building was instead configured for other missions. The first active mission was the Actinide Billet Line (ABL) which made targets that would eventually end up as Pu-238 pellets for NASA deep space missions. The second mission was the Plutonium Experimental Facility (PEF), which was started alongside the Plutonium Fuel Form (PuFF) Facility, both of which focused on the production of Np-237 as sources of Pu-238 fuel pellets for thermal generation. This process eventually included the Old Metallurgical Laboratory (OML) that focused on qualification testing of the pellets generated in the PuFF Facility for deep space craft.[11]

Building 235-F is a windowless, reinforced concrete structure approximately 222 feet long, 109 feet wide and 28 feet high. The two-story building was designed and built as a blast-resistant structure and has double reinforced 14-inch-thick exterior walls. Pier footings and columns support the 8-inch second floor and roof slabs which are directly supported by a reinforced concrete beam and girder system. Within the building, exhaust air from various process areas/enclosures is passed through double High Efficiency Particulate Air (HEPA) filtration before discharge. Various other buildings and ancillary structures supported operations within the 235-F complex.[11]

Nuclear production processes were located on both the first and second floors. Primary support functions were located on the second floor, with storage vaults located on both the first and second floors. Physical barriers separate the building into clean areas and Radiologically Controlled Areas (RCA). Each of the areas is served by separate once-through ventilation systems consisting of supply and exhaust fans operating in a "push-pull" configuration, with multiple airlocks between processing areas and entrances into the building. Air locks are installed at interfaces between clean areas and RCAs to allow personnel to move through the building without requiring the entire facility to be an RCA, while also providing additional confinement of radiological material from the processing areas.

As the metallurgical facility for F-area, Building 235-F was meant to fabricate the separated plutonium metal into weapons components.[11] However, before any equipment was installed, the mission was cancelled and relocated to Rocky Flats Plant. As such, 235-F was reconfigured for other missions.[15] There were four primary process areas located within 235-F building during operational periods. During the operational life of building 235-F the production processes were primarily dry mechanical fabrication.

- The ABL was used most recently to fabricate billets from Np-237 oxide powder for extrusion into reactor targets for production of Pu-238 in support of radioisotope thermoelectric generators for deep space missions. Previously, ABL also ran targets containing Pu-239 in support of the weapons mission. Remaining contents consist mainly of a glovebox line and its associated equipment that has undergone some decontamination. The ABL continued operations until 1991. The gloveboxes are maintained at negative pressure by the process exhaust ventilation system for confinement of the residual material.

- The PuFF Facility produced Pu-238 heat source pellets and contained a hot cell train of 9 process cells and associated infrastructure behind the cells (entry hoods for moving material into the hot cells, gloves for manual manipulation, etc.). Manipulation of material/equipment was performed predominantly by master-slave manipulators. Pu-238 was processed in the PuFF Facility from 1977 until 1983. The process involved ball milling of fine Pu-238 into ultrafine particulate for subsequent metallurgical pressing, coating in iridium, and decontamination for removal from the hot cells. The process resulted in significant powder contamination within the hot cells in the PuFF Facility. The cells and associated gloveboxes are maintained at negative pressure by the process exhaust ventilation system for confinement of the residual material.

- PEF activities included research and development (R&D) on processes that manufactured Pu-238 heat sources. Pu-238 was processed in the PEF to develop the fuel pellet fabrication process from 1979 to 1981. The Pu-238 inventory was removed when the facility was shutdown. To ensure safety, the glovebox line is maintained at negative pressure by the process exhaust ventilation system for confinement of residual plutonium oxide.

- The OML tested Pu-238 heat source pellets for compliance to design specifications prior to shipment for inclusion into deep space crafts. The glovebox line is maintained at negative pressure by the process exhaust ventilation system for confinement of residual plutonium oxide contamination.

The ABL, OML, and PEF continued operations until SRS production support within the facility was shut down in 1991. The PuFF process area operated until 1983 when it was transitioned to warm standby until subsequent shut down in 1991.[11] The building's last mission provided for the receipt, storage (within vaults), and disbursement of plutonium bearing materials in support of SRS and the DOE Complex. In 2006, the vaults were de-inventoried, and the facility was transitioned from operation to a reduced S&M state through 2012. Various deactivation activities occurred in Building 235-F between 2013 and 2018 with a focus on using traditional approaches to remove residual hold-up and MAR. Scoping for the decontamination of PuFF cells was originally limited to proven methods. In 2015, SRNL and FIU began collaboration on a project to address waste and D&D engineering and technology development specifically focused on the application of incombustible fixatives to reduce the risk of residual radioactive contamination, with a target of using the 235-F PuFF cells and entry hoods as a basis for development.

2.2.2 PuFF Challenges

Though the PuFF Facility inside 235-F poses many of the challenges of D&D in any radiologically contaminated facility, there are additional challenges due to configuration and contamination. There are only single penetrations 12 in (30.5 cm) in diameter with airlock doors on each side. In addition to the limited penetration points which prevent easy maneuverability, the manipulators that were installed from prior processing have succumbed to mechanical degradation from the incident alpha particle bombardment from the Pu-238, resulting in significant aluminum powder and hydraulic grease on the floor of most cells This restricts decontamination efforts to the gloves installed at the back of the cells, requiring the use of long handled tools for much of the manipulation and significant dose rates from some cells to workers. Maintaining confinement during setup and maintenance is also challenging due to the configuration and contamination with Pu-238.

The Pu-238 contaminant is known to be fine particulate due to the singular mission space of the PuFF hot cells, and is distributed in the cells as airborne, fixed, and transferable contamination. The Pu-238, primarily as plutonium oxide, resides on all surfaces of the cells as a non-visible layer with more accumulation at low points near or on the floors of the cells. Due to self-heating and alpha recoil, a large portion of the material is thought to be submicron particles.[10] Because of the submicron particle size, the contamination is very mobile, can move from its initial location, and is likely to remain suspended for days. Because of this high mobility, loss of confinement of the material inside the cells is of significant concern, as are the potential impacts of postulated accident scenarios in the BIO that would lead to significant breach of these containment structures. Any technological solution intended to reduce the risk associated with residual contamination at the facility, to include fixatives, will need to adequately address these conditions and additional considerations including the building having existed in extended S&M for 30 years as of this report.

3.0 Incombustible Fixative Research Activity in Support of SRS 235-F

In discussions among representatives from SRS, DOE EM, SRNL, and FIU, the requirement for enhancing the operational performance of fixatives to better address the unique D&D challenges faced by the SRS 235-F Project and the DOE EM Complex at large, was identified as a priority focus area for knowledge building and R&D. Existing technologies, and their respective application methods, have had varying degrees of effectiveness, possessing strengths in certain areas and opportunities for improvement in others. Of particular interest was the operational requirement for a fixative to continue to immobilize a residual contaminant even when exposed to the extreme thermal stressors and other challenging environmental conditions postulated in various accident scenarios in BIOs and safety basis documents, especially where plutonium is the contaminant of concern.

The most common risk scenario identified across the DOE Complex that threatened D&D activities is fire loading. Fire represents a postulated risk due to several internal factors such as: degraded electrical equipment, antiquated fire suppression systems, and lack of viable fire barriers. External factors also contribute to fire risk, including seismically-induced fires and other natural disasters. A worst-case scenario of a seismically-induced full-facility fire was determined to be greater than 10 rem off-site and 27,000 rem to workers at 100 meters at the SRS 235-F PuFF Facility. Proactive fire prevention control measures are implemented at SRS, including the elimination of all potential ignition sources, controlling the amount of combustibles in the facility, and maximum removal of contamination. That said, the stakeholders are exceptionally proactive in identifying tools and other technologies that are inherently fire resilient and can reduce the risk of release of residual contamination when exposed to extreme thermal stressors. This proactivity has not historically considered the risk of a flammable fixative application, however, which could provide a new pathway for flame propagation as well as increasing the overall combustible loading of the facility.

To address these issues, SRNL and FIU Applied Research Center (ARC) funded through DOE EM led the implementation and execution of a phased research activity to improve the operational effectiveness of fixative technologies in the critical area of fire resistance. Baselining of current industrial fixatives encompassed **Phase I** of this research project. After baselining current industry fixatives, research was conducted into fire resilient technologies in other industries. It was hypothesized that Intumescent Coatings (IC) could potentially operate as a fire-resistant layer or as a standalone fixative for use in the D&D space. Initially developed to protect and insulate various substrates from extreme heat and fire conditions in order to maintain their structural integrity, research revealed that in certain instances the fire protection was so effective that it protected the primer itself on the substrate.[16] **Phase II** encompassed the proof-of-concept of IC technologies as a potential viable solution, and the subsequent down-selection of a specific technology was made. Comprehensive test evaluation and analysis of the down-selected intumescent coating was conducted, and a cold test was carried out using mock-ups of a typical process cell both at FIU and SRS during **Phase III**. The cold demo explored different application techniques and tools required to apply intumescent coatings to the mockup process cell, and generated crucial operational lessons learned for transitioning to the real environment. After cold demos were completed, a hot test and post-application testing where carried out in 235-F. The hot test and post-application testing comprised **Phase IV** of this research.

4.0 Phase I: Baselining Industry Fixatives Commonly Used Across the DOE EM Complex

Phase I of this research activity was to ascertain how well existing fixative technologies commonly used across the DOE EM Complex perform when exposed to thermal and environmental stressors. Determining the current state of the art was deemed an essential element of this effort, particularly since a comprehensive assessment of current fixative technologies, under these conditions, had not been conducted to date. Furthermore, establishing a thermal stressor baseline for the current fixative products would facilitate future analysis in quantifying improvements in operational performance of alternative technological solutions being hypothesized (e.g.: use of intumescent technologies). Studies of baseline behavior of four (4) commonly used fixatives and one (1) decontamination gel were performed, and notable shortfalls and deficiencies were revealed, particularly in the critical area of postulated fire scenarios as outlined in facility BIOs. The decontamination gel was included as historic use of these types of materials have occasionally been substituted as fixatives. Additional vulnerabilities were identified during the environmental testing by SRNL, with higher relative humidity and water immersion negatively impacting adhesion and performance.

It is important to highlight a common theme that will be reiterated throughout this technical report, specifically that there were no existing formal standard performance specifications or uniform fire / thermal stressor testing protocols for D&D fixatives. Therefore, FIU ARC and SRNL researchers decided to develop a series of tests that incorporated various elements of certain American Society of Testing and Materials (ASTM) International fire-related standards designed for paints, coatings, and construction materials. ASTM D1360 (Standard Test Method for Fire Retardancy of Paints), ASTM E119 (Standard Test Methods for Fire Tests of Building Construction and Materials), and ASTM E84 (Standard Test Method for Surface Burning Characteristics of Building Materials) were chosen to serve as “near fit” protocols. In collaboration with the various stakeholders, those elements deemed most appropriate from the above-mentioned ASTM standards were compiled and modified where necessary for the experimental design related to thermal stressor testing.

4.1 Selected Industry Fixatives

The following commonly used industry fixatives were evaluated:

1. Product A: Product A is a high solids asbestos encapsulant/sealant, designed to encapsulate friable asbestos-containing material (ACM) such as fireproofing and insulation material. The high solids, nonflammable composition of Product A allows for dilution with water to provide maximum flexibility for specific asbestos abatement needs, including lockdown/removal, penetrating encapsulation, and bridging encapsulation. It forms a barrier between hazardous or contaminated materials and the environment.
2. Product B: Product B is a non-toxic, water-based solution which forms an impermeable barrier between hazardous or contaminated materials and the environment. It can be applied to any surface to lock down loose contamination and prevent leaching of contaminates after decontamination efforts. It is commonly used to stabilize large plant components, concrete, valves, and other problematic radioactive waste equipment prior to shipment.
3. Product C: Product C is an epoxy that permanently stabilizes radiological particulates, beryllium, asbestos, and other hazardous contamination. It is used as the final step in a two-step approach to permanently fix contamination on surfaces. When part B of Product C is applied over loose contamination (previously sprayed with part A of Product C), the dust and debris is re-hydrated and absorbed into the matrix of the fixative. The result of this process is a hard coating that ensures that stabilization of all dust and debris has been achieved.
4. Product D: Product D is recommended for decontamination of radioisotopes as well as particulates, heavy metals, water-soluble and insoluble organic compounds, including tritiated compounds. The

hydrogel coating can be applied to horizontal, vertical and inverted surfaces and can be applied to most materials including bare, coated and painted concrete, aluminum, steel, lead, rubber, Plexiglas, herculite, wood, porcelain, tile grout, and vinyl, ceramic and linoleum floor tiles. When dry, the product locks the contaminants into a polymer matrix. The film containing the encapsulated contamination can then be peeled and disposed of according to appropriate local, state, and federal regulations.

5. Product E: Product E is a heavy-duty concrete and steel floor system that provides a high gloss, seamless, durable surface in nuclear facilities subject to radiation, decontamination and loss of coolant accident.

4.2 Experimental Design Summary for Exposure to Thermal Stressors

Researchers subjected the fixatives to two forms of thermal stressor. The first of these entailed exposure to ever increasing temperatures for a specified period in a muffle furnace, and the second was exposure to a direct flame.

4.2.1 Muffle Furnace Test

In order to baseline the selected fixative's resiliency against extreme heat, FIU ARC applied each of the fixatives to 4 in x 4 in x 1/84 in (10.16 cm x 10.16 cm x 0.03 cm) stainless steel coupons in accordance with the manufacturer's instructions, and then exposed them to incremental 100 °F (37.78 °C) temperature increases from 200 °F – 800 °F (93.33 °C – 426.67 °C) for a period of 15 minutes at each temperature setting. Data points were observed and recorded (visual observations and mass measurements) at the onset as well as at the completion of each temperature setting with the intent of developing mass loss profiles for each fixative. To further demonstrate the operational impacts of extreme heat on the respective fixatives, FIU ARC used Glo Germ to simulate a contaminant. Glo Germ is a non-toxic, temperature stable powder simulant that will fluoresce when exposed to ultraviolet (UV) light and is often used to simulate contamination spread. The Glo Germ was coated on the stainless steel coupon, with the selected fixative then being applied over it to immobilize the simulated contaminant (Figure 4-1). The coupon was then subjected to the same pattern of incremental temperature increases for the same time periods as outlined above. Any runoff that may have occurred due to melting of the fixative was captured in a Petri dish placed underneath the coupon.

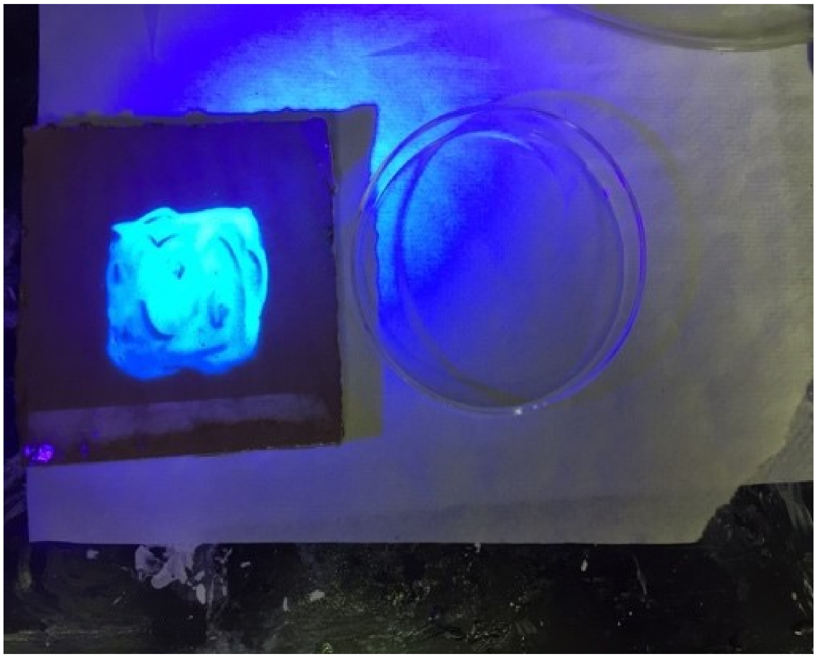
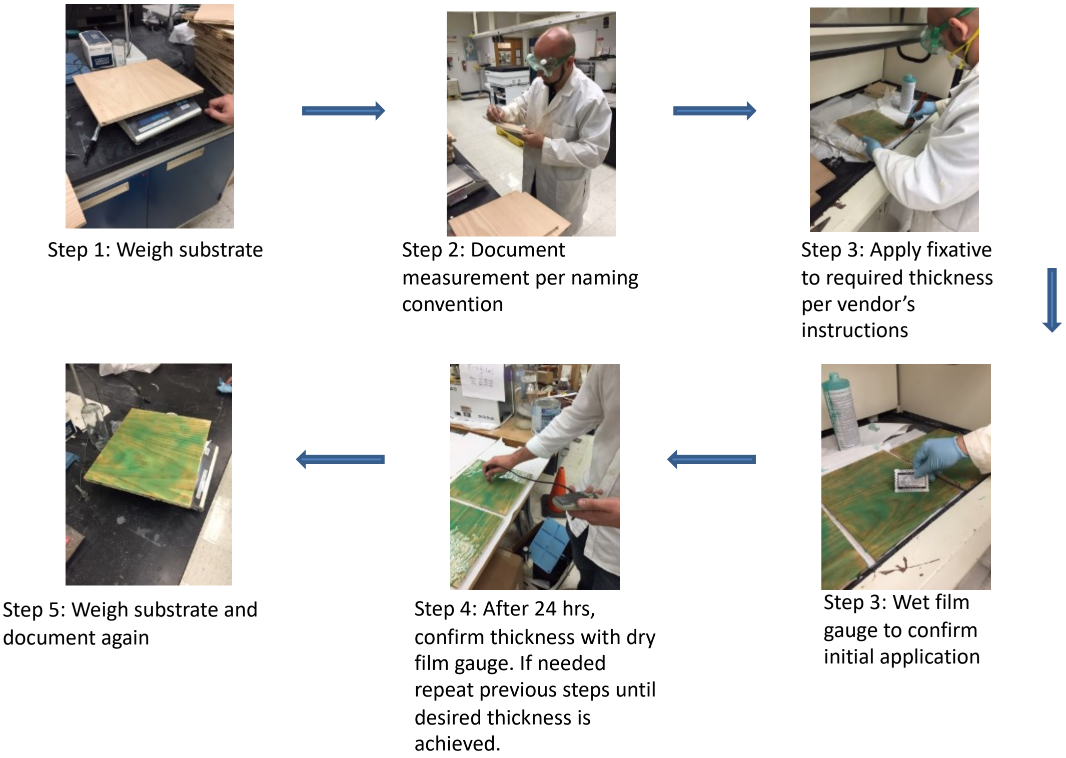


Figure 4-1. Glo Germ plus fixative applied to test coupon.

4.2.2 Open Flame Test

To test fire retardant characteristics when exposed to an open flame, ARC constructed an apparatus which allowed for the testing of three (3) test coupons simultaneously. The designated fixatives were applied to a 12 in x 12 in x ½ in (30.48 cm x 30.48 cm x 1.27 cm) red oak coupon in strict accordance with the manufacturer's instructions to the requisite thickness, under ideal environmental conditions (47% humidity and 72 ºF (22.22 ºC)), and were then allowed to cure for the manufacturer specified time (Figure 4-2). Once the curing process was complete on the designated test coupons, relevant observations were made and documented in terms of visual observations, total mass, and fixative thickness. They were then placed in the open flame testing apparatus and exposed to a flame generated by a propane torch placed perpendicular to the sample approximately 4 in (10.16 cm) from the center of the coupon for up to 5 minutes, depending on the resiliency of the fixative (Figure 4-3). Observations were noted and data collected.



Step 1: Weigh substrate

Step 2: Document measurement per naming convention

Step 3: Apply fixative to required thickness per vendor’s instructions

Step 3: Wet film gauge to confirm initial application

Step 4: After 24 hrs, confirm thickness with dry film gauge. If needed repeat previous steps until desired thickness is achieved.

Step 5: Weigh substrate and document again

Figure 4-2. Open flame test coupon preparation procedure for fixatives.

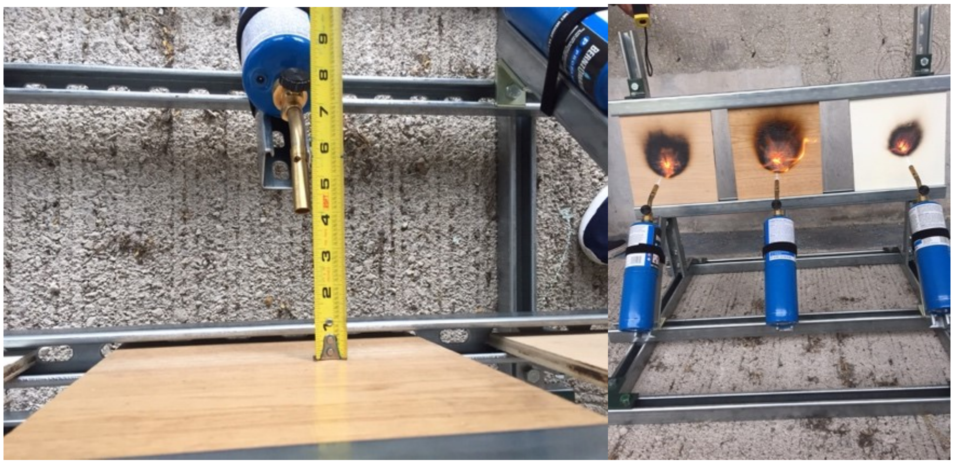


Figure 4-3. Open flame testing apparatus with fixative-coated red oak test coupons.

4.3 Thermal Stressor Testing Results Summary[16]

As highlighted in the Executive Summary, the series of baseline experiments implemented by ARC for industry fixatives currently in use identified significant shortfalls and deficiencies in their fire-retardant characteristics. The highlights below outline some of the key findings from ARC's baseline testing, with the associated table and figures providing a more detailed depiction of the actual impacts:

1. At temperatures as low as 300-400 °F (~149-204 °C), melting from the substrate occurred with every fixative/decontamination gel in as little as 3-5 minutes of exposure, resulting in contaminant transport (Figure 4-5).
2. All 5 fixatives/decontamination gels began to exhibit mass loss at temperatures as low as 200°F (149 °C), and the most significant degradation in terms of mass loss, desiccation, off-gassing, and chemical breakdown occurred between 600-800 °F (~315-427 °C).
3. All 5 fixatives/decontamination gels lost between 70% to 90% mass when exposed to fifteen-minute incremental temperature increases between 200-800 °F (149-427 °C). The breakdown was so complete that researchers assessed there would likely have been a release of radioisotopes if applied to a contaminated substrate.
4. All 5 fixatives/decontamination gels ignited/became flammable almost immediately when exposed to an open flame and burned completely off the various substrates within 1-5 minutes. A likely release of radioisotopes was deduced if applied to a contaminated substrate. This also supports the thought that these materials could serve as a propagation method of flame if applied to non-flammable surfaces such as concrete or stainless steel.

Photo documentation for each individual fixative at various stages of the muffle furnace testing is contained below, but researchers were able to compose a "Basic Fixative Profile" based on the data (Figure 4-4). It depicts the progression of degradation and compounding negative impacts at increasing temperatures over the 2-hour period. In every instance, the respective fixative being tested began to melt at temperatures as low as 300°F, causing our simulated contaminant (Glo Germ) to flow freely off the substrate, thereby failing to fix/immobilize the contaminant when exposed to this type of thermal stressor, representing a potential release. Its overall performance continued to degrade, with each fixative losing between 75%-95% of its mass, becoming a shell of itself at 800 °F (427 °C).

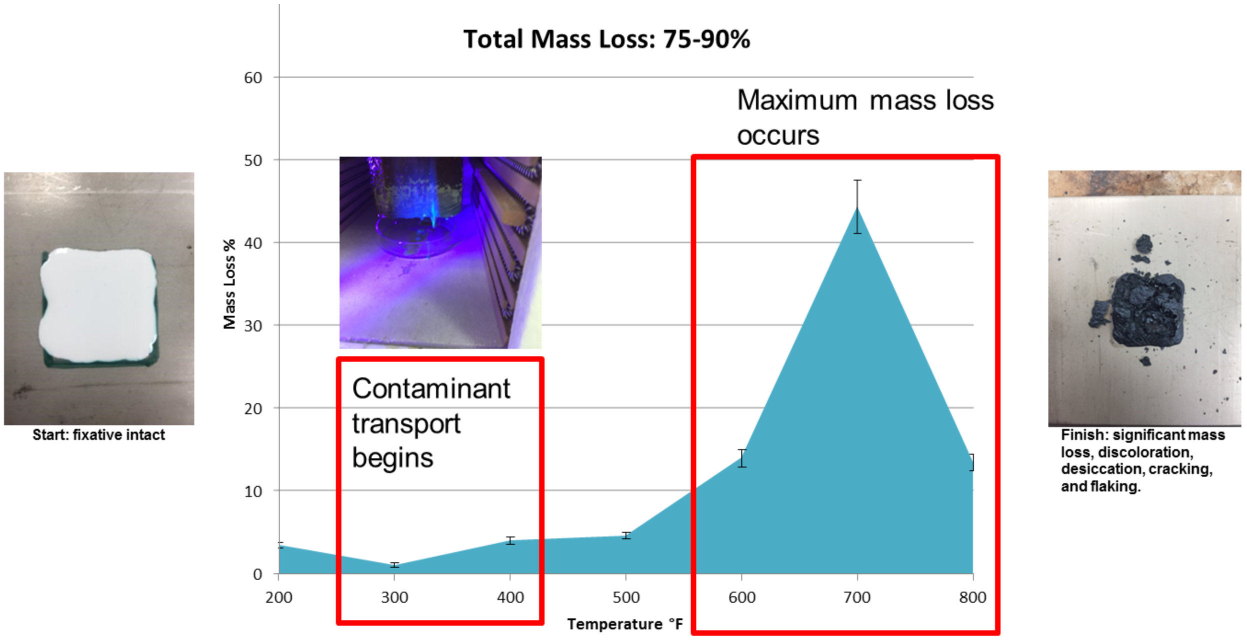


Figure 4-4. Basic fixative profile when exposed to extreme heat.

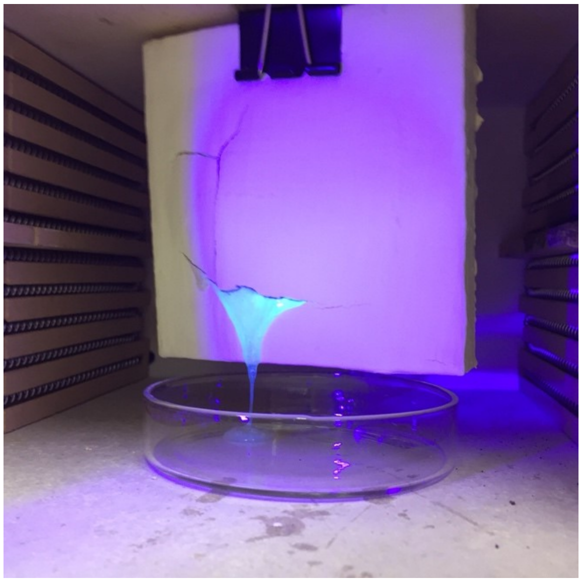
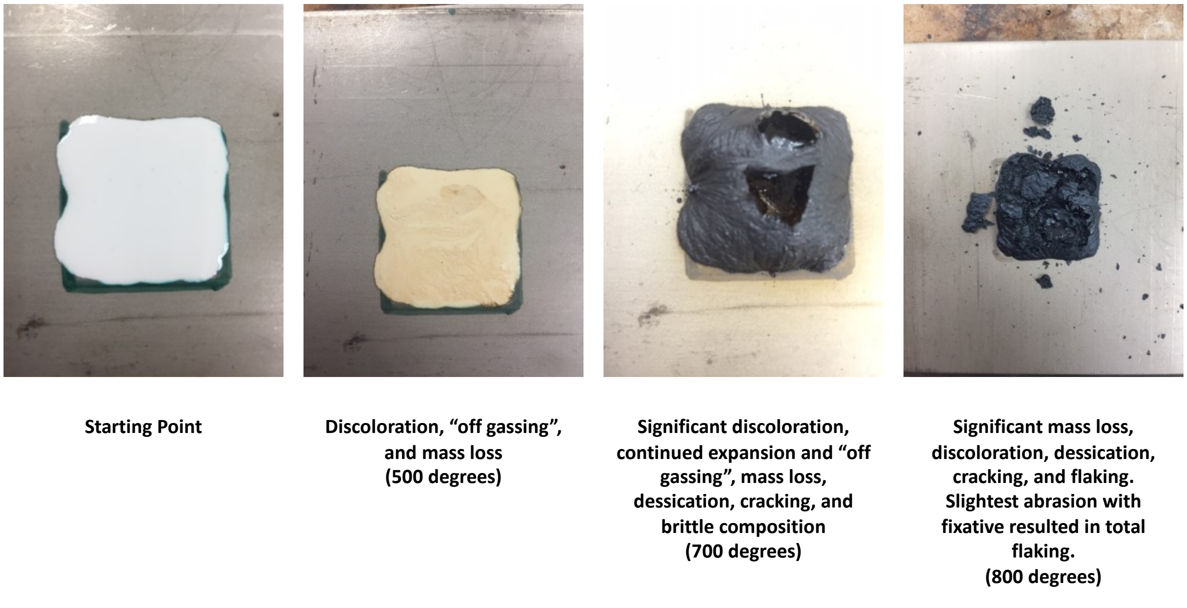


Figure 4-5. Glo Germ flowing at 300 °F with Fixative C.

As depicted above, the use of Glo Germ, a UV fluorescing compound, as a simulated contaminant allowed ARC researchers to identify the general impacts on contaminant flow when the fixatives were exposed to increasing temperatures. In every instance, for all 5 fixatives baselined, contaminant flow was observed between 250 °F (121 °C) and 450°F (232 °C). Furthermore, each fixative baselined displayed characteristics indicative of a near complete structural breakdown at temperatures between 600 °F (315 °C) and 800 °F (427 °C). Finally, during the open flame series of baseline tests, all 5 fixatives demonstrated notable deficiencies, with each one burning completely off the substrate within 1-5 minutes.



Starting Point

Discoloration, "off gassing", and mass loss (500 degrees)

Significant discoloration, continued expansion and "off gassing", mass loss, dessication, cracking, and brittle composition (700 degrees)

Significant mass loss, discoloration, dessication, cracking, and flaking. Slightest abrasion with fixative resulted in total flaking. (800 degrees)

Figure 4-6. Observed Impacts to Fixative "E".

Figure 4-7. Fixative B burning off a red oak substrate test coupon.

Figure 4-8. Fixative A continuing to burn after removal of flame source.

4.4 Environmental Stressor Testing and Adhesion Impacts Summary[13]

4.4.1 Non-ideal Environmental Effects on Curing Time and Adhesion

To determine the performance of these fixatives in non-ideal environments, sample stainless steel coupons coated with the selected fixatives were subjected to various temperatures and relative humidities (RHs) pre-cure, prepared per the manufacturer's specifications. In all cases, a maximum of 8 hours was chosen as a maximum due to operational concerns associated with an 8-hour cure time deeming the material unattractive for use in the facility. The testing monitored:

- Set-to-touch time – the time it takes for the fixative to be considered functional without full curing. This was tested by touching the surface with a finger in a nitrile glove and rolling the finger across a glass slide. The sample was considered set to touch when the glass showed no residue had been transferred.

- Dust-free time – the time it takes for the fixative to no longer collect and/or attract ambient material on the external surface. This was tested by placing a small amount of cotton on the surface and gently blowing parallel to the substrate surface. The sample was considered to pass once the cotton could be blown gently off the surface.

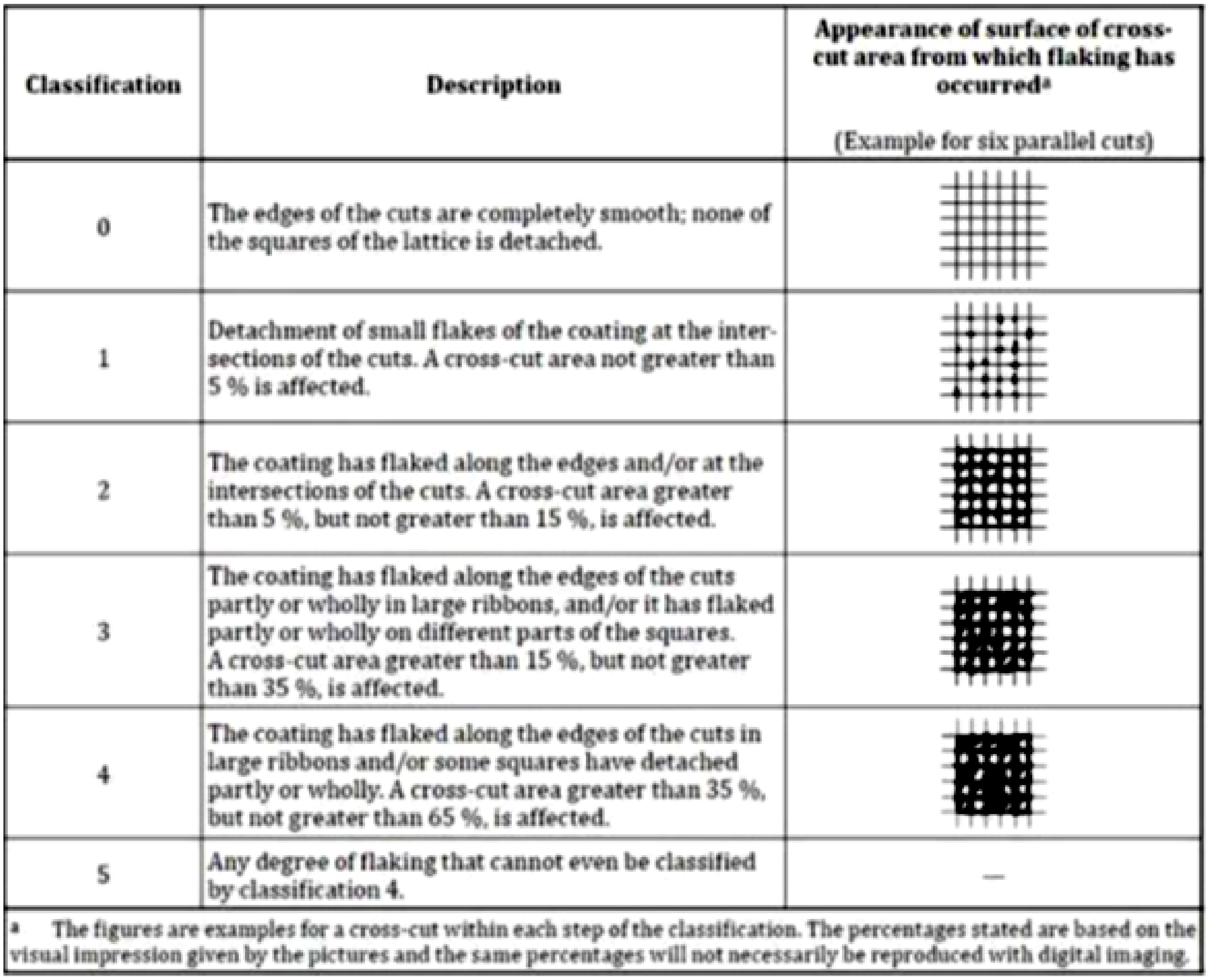
- Dry-to-touch – the time it takes for the fixative to be considered fully cured. This was tested by touching the substrate gently with nitrile gloves and was considered dry to touch when the material no longer adhered to the finger and did not rub up appreciably when the finger is lightly rubbed across the surface.

- Adhesion – how well the fixative sticks to the substrate after full curing. This was tested using the cross hatch method described below.

Adhesion testing was performed per ASTM D3359 once curing was achieved.[17] Using the cross-hatch tool (Figure 4-9), an “X” pattern was sliced into the cured coating, and the resulting material left on the substrate was compared to the diagram shown in Table 4-1.

Figure 4-9: Cross-hatch Tool for Adhesion Testing per ASTM D3359.[17]

Table 4-1: Cross-hatch Scale with Reference Diagrams.[17]



Classification	Description	Appearance of surface of cross-cut area from which flaking has occurred[a] (Example for six parallel cuts)
0	The edges of the cuts are completely smooth; none of the squares of the lattice is detached.	
1	Detachment of small flakes of the coating at the intersections of the cuts. A cross-cut area not greater than 5 % is affected.	
2	The coating has flaked along the edges and/or at the intersections of the cuts. A cross-cut area greater than 5 %, but not greater than 15 %, is affected.	
3	The coating has flaked along the edges of the cuts partly or wholly in large ribbons, and/or it has flaked partly or wholly on different parts of the squares. A cross-cut area greater than 15 %, but not greater than 35 %, is affected.	
4	The coating has flaked along the edges of the cuts in large ribbons and/or some squares have detached partly or wholly. A cross-cut area greater than 35 %, but not greater than 65 %, is affected.	
5	Any degree of flaking that cannot even be classified by classification 4.	—

[a] The figures are examples for a cross-cut within each step of the classification. The percentages stated are based on the visual impression given by the pictures and the same percentages will not necessarily be reproduced with digital imaging.

Summary tables (Table 4-2–Table 4-5) for each of the materials are given below, with highlights on significant differences from the baseline test at 75 °F (24 °C) and 40% RH. Other comments on the fixatives are given on a sample-by-sample basis with the charts. Overall, greater variation in cure times was assigned more to the humidity for the non-epoxy fixatives, with epoxy fixatives showing sensitivity to both humidity and temperature. Samples that were unable to cure at a given temperature/humidity range are denoted with an "X" in the column for tests that were never passed in the 24-hour span. Samples that experienced significant increases in respective testing times (≥30 minutes) are shaded red and those that experienced significant decreases in threshold times (≥30 minutes) are shaded blue. The greatest correlation to the non-epoxy fixatives were found in the humidity of the environment, while the epoxy materials showed strong dependence on both temperature and humidity. For epoxy materials, this dependence was so great that it became impossible to cure them in some of the tested environments. Product E was eliminated as a viable candidate prior to this testing due to complete delamination from wood and stainless steel substrate, and as such no results are reported for that material.

Table 4-2. Summary data for Product A

Product A	**75 ° F/ 40% RH**	**20 ° F/ 0% RH**	**40 ° F/ 40% RH**	**60 ° F/ 40% RH**	**90 ° F/ 40% RH**	**110 ° F/ 40% RH**	**75 ° F/ 10% RH**	**75 ° F/ 20% RH**	**75 ° F/ 60% RH**	**75 ° F/ 80% RH**	**75 ° F/ 95% RH**
Set to Touch (hr)	1:00	1:00	1:00	1:00	1:00	1:00	1:00	1:00	1:00	2:00	3:30
Dust Free (hr)	1:30	2:00	1:30	1:30	1:30	1:30	1:30	1:30	1:30	2:30	4:00
Dry to Touch (hr)	1:30	2:00	1:30	1:30	1:30	1:30	1:30	1:30	1:30	2:30	4:00
D3359	Class 0	Class 0	Class 5	Class 1	Class 5	Class 2	Class 1	Class 5	Class 5	Class 2	Class 5
Average thickness (mm)	0.531	0.341	0.529	0.496	0.105	0.559	0.685	0.508	0.144	0.589	0.216

Comments: High humidity (≥80% RH) caused significant changes in cure times, minor changes at low (20 °F) temperatures. Variation in D3359 tests are due to chips that occasionally delaminate during testing.

Table 4-3. Summary data for Product B

Product B	**75 ° F/ 40% RH**	**20 ° F/ 0% RH**	**40 ° F/ 40% RH**	**60 ° F/ 40% RH**	**90 ° F/ 40% RH**	**110 ° F/ 40% RH**	**75 ° F/ 10% RH**	**75 ° F/ 20% RH**	**75 ° F/ 60% RH**	**75 ° F/ 80% RH**	**75 ° F/ 95% RH**
Set to Touch (hr)	1:00	1:00	1:00	1:00	1:00	1:00	1:00	1:00	1:00	1:00	3:30
Dust Free (hr)	1:30	1:30	1:30	1:30	1:30	1:30	1:30	1:30	1:30	1:30	4:00
Dry to Touch (hr)	1:30	1:30	1:30	1:30	1:30	1:30	1:30	1:30	1:30	1:30	4:00
D3359	Class 0	Class 4	Class 0	Class 1	Class 0	Class 0	Class 0	Class 0	Class 1	Class 1	Class 0
Average thickness (mm)	1.04	0.553	0.341	0.408	0.322	0.446	0.581	0.432	0.413	0.320	0.297

Comments: Changes to the curing time were notable at the extreme ends of humidity. ASTM D3359 tests showed a significant difference during the low temperature cure test signifying an impact on adhesion.

Table 4-4. Summary data for Product C

Product C	**75 ° F/ 40% RH**	**20 ° F/ 0% RH**	**40 ° F/ 40% RH**	**60 ° F/ 40% RH**	**90 ° F/ 40% RH**	**110 ° F/ 40% RH**	**75 ° F/ 10% RH**	**75 ° F/ 20% RH**	**75 ° F/ 60% RH**	**75 ° F/ 80% RH**	**75 °F / 95% RH**
Set to Touch (hr)	3:00	X	8+	4:00	1:30	1:00	4:30	2:30	2:00	2:30	3:30
Dust Free (hr)	3:00	X	8+	7:00	2:00	1:30	5:00	4:00	4:30	3:00	4:00

Dry to Touch (hr)	3:30	X	8+	7:00	2:00	1:30	5:00	4:00	4:30	3:00	4:00
D3359	Class 1	X	Class 0	Class 0	Class 0	Class 0	class 0	Class 0	Class 0	Class 0	Class 0
Average thickness	1.015	X	1.074	0.790	0.487	0.853	0.705	0.450	0.553	1.001	0.605

Comments: Product C demonstrated a greater dependence on the curing temperature over that of relative humidity. Along with the other two epoxies, it is impossible for the material to cure at 20 °F.

Table 4-5. Summary data for Product D

Product D	**75 °F/ 40% RH**	**20 °F/ 0% RH**	**40 °F/ 40 % RH**	**60 °F/ 40 % RH**	**90 °F/ 40 % RH**	**110 °F/ 40% RH**	**75 °F/ 10% RH**	**75 °F/ 20 % RH**	**75 °F/ 60 % RH**	**75 °F/ 80 % RH**	**75 °F/ 95% RH**
Set to Touch (hr)	1:00	1:00	1:00	1:00	1:00	1:00	1:00	1:00	1:00	2:00	2:30
Dust Free (hr)	1:30	1:30	1:30	1:30	1:30	1:30	1:30	1:30	1:30	2:30	3:00
Dry to Touch (hr)	1:30	1:30	1:30	1:30	1:30	1:30	1:30	1:30	1:30	2:30	3:00
D3359	Class 0	Class 0	Class 5	Class 0	Class 5	Class 0	Class 0	Class 5	Class 5	Class 5	Class 5
Average thickness (mm)	0.269	0.142	0.114	0.080	0.063	0.173	0.171	0.092	0.135	0.087	0.109

Comments: High humidity (≥80% RH) caused moderate changes in cure times, D3359 testing either caused significant delamination of the thin film from the substrate or had no effect with no correlation between temperature or relative humidity.

4.4.2 Non-ideal Environmental Effects on Post-cured Fixatives[13]

To determine the behavior of the fixatives in non-ideal environmental conditions anticipated of a facility in extended S&M, sample coupons were prepared on stainless steel, wood, and sheet rock. These substrates were chosen as they are the most commonly found materials for building surfaces. It should be noted wood and sheet rock are not typical in US nuclear facilities, however, they are routinely found in international nuclear facilities, validating the need for these data sets. Multiple environmental conditions were chosen to simulate conditions that might be found in a nuclear facility after environmental control is disabled. To ensure variables could be isolated, two exposure profiles were tested: variable temperature at a static 40% RH, and variable humidity at a static 75 ºF. The temperatures chosen for this experiment were: 20, 40, 60, 75, 90, and 110 ºF (-7, 4.5, 15.5, 24, 32, and 43 ºC). The humidities chosen for this experiment were: 10, 20, 40, 60, 80, and 95% RH. 10 and 95% were chosen as bounds as these were the limits of the environmental chamber used. Samples from all sets (sheetrock, wood, and stainless steel) were placed in the environmental chamber as shown in Figure 4-10 and exposed to the chosen environmental condition for 48 hours. Blanks were included as controls to eliminate substrate bias. It should be noted that the below figure shows six (6) different fixatives being tested, however one of the products was immediately disqualified due to complete delamination from stainless steel and wood upon receipt. It was determined to test the delaminated film on top of the substrate to determine any impacts on the delaminated film, however after initial testing, this fixative was completely excluded from other data sets, and as such is not discussed in this document.

Post-cure results were observed to cluster in three groups: at 20 ºF (-7 ºC), above 75 ºF (24 ºC), and above 40% RH.

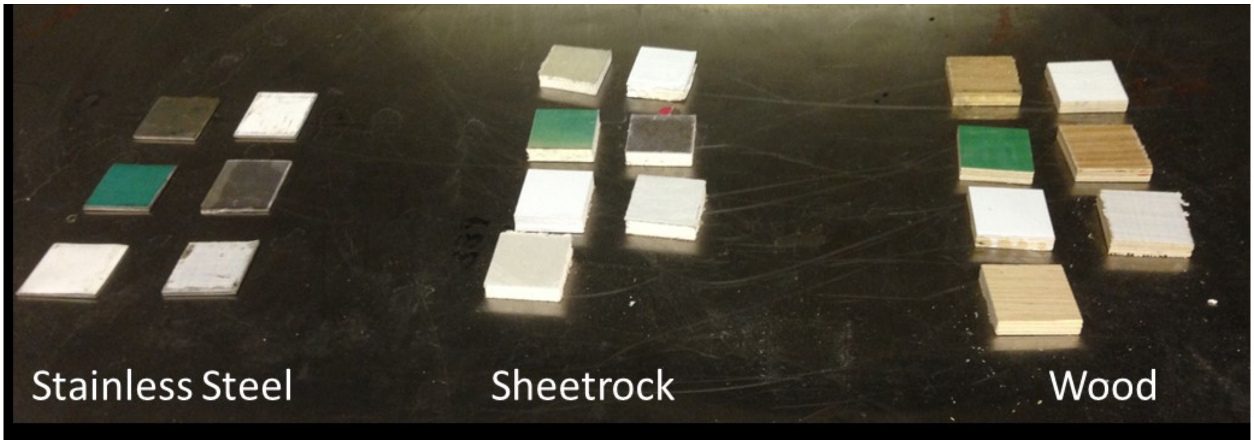


Figure 4-10: Environmental Chamber Setup of Post-cured Samples.

4.4.2.1 20 ºF (-7 ºC) Response

The only fixative to show response to low temperatures was Product B on the wood and sheetrock surfaces. For this testing, both showed heavy delamination, with the sheetrock delamination also taking the first layer off the substrate. All other materials proved unaffected in this regime of testing, maintaining their coating without any denaturing.

4.4.2.2 Above 75 ºF (24 ºC) Response

Product A proved susceptible to elevated temperatures on both metal and wood substrates, with a partial melting off the substrate and onto the environmental chamber floor occurring at 110 ºF. The material re-solidified after cooling. The sheetrock coated with Product A proved to be visually and tactilely unaffected. Product C also showed a partial melting similar to Product A at 110 ºF (43 ºC) for both the metal and wood substrates while the sheetrock showed to be unaffected. The samples deposited on metal surfaces were more dramatically affected and caused a melt off the metal substrate whereas the wood substrate only showed running of the fixatives down the edges after temperature cycling. Product D showed partial delamination that became more apparent as the temperature increased above 75 ºF (24 ºC) with the entire polymer sheet becoming delaminated at 110 ºF (43 ºC) on metal and running down the side of the wood substrate. The sheetrock sample was also affected and showed a slight run down the edge of the sample and partial delamination from the substrate. Product B was unaffected in this regime of testing, maintaining the coating without any denaturing.

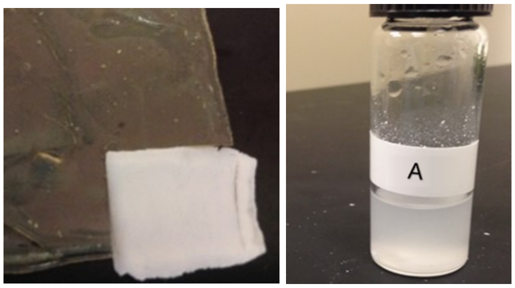
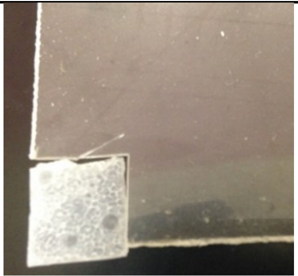
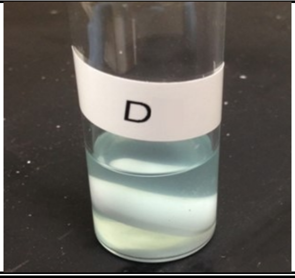
4.4.2.3 Above 40% RH

Product A was only affected by high humidities (≥80% RH) on the wood substrate, exhibiting swelling and wetness to the touch after the humidity cycling was complete. The metal and sheetrock samples were unaffected. This is most likely due to the porosity of wood allowing for water to interface with the material better while the sheetrock sample simply absorbs the moisture into the substrate. Product C only responded to high humidities (≥60% RH) on the sheetrock substrate, heavily delaminating from the substrate, peeling away the exterior layer of sheetrock, and exhibiting the cracking noted during the water solubility/response testing. The wood and metal substrates proved unaffected. Product B showed humidity susceptibility on both the metal and sheetrock substrates. For the metal substrate, water retention was apparent for ≥80% RH due to tactile swelling and reduction of surface tackiness on par with the submerged sample, while the sheetrock sample showed delamination akin to the Product C in this regime. The Product B wood sample was unaffected by high humidities. Product D for all substrates showed high water retention at ≥60% RH and showed partial dissolution/running of the sample off the substrate for humidities ≥80% RH.

4.4.3 Water Solubility Testing Summary[13]

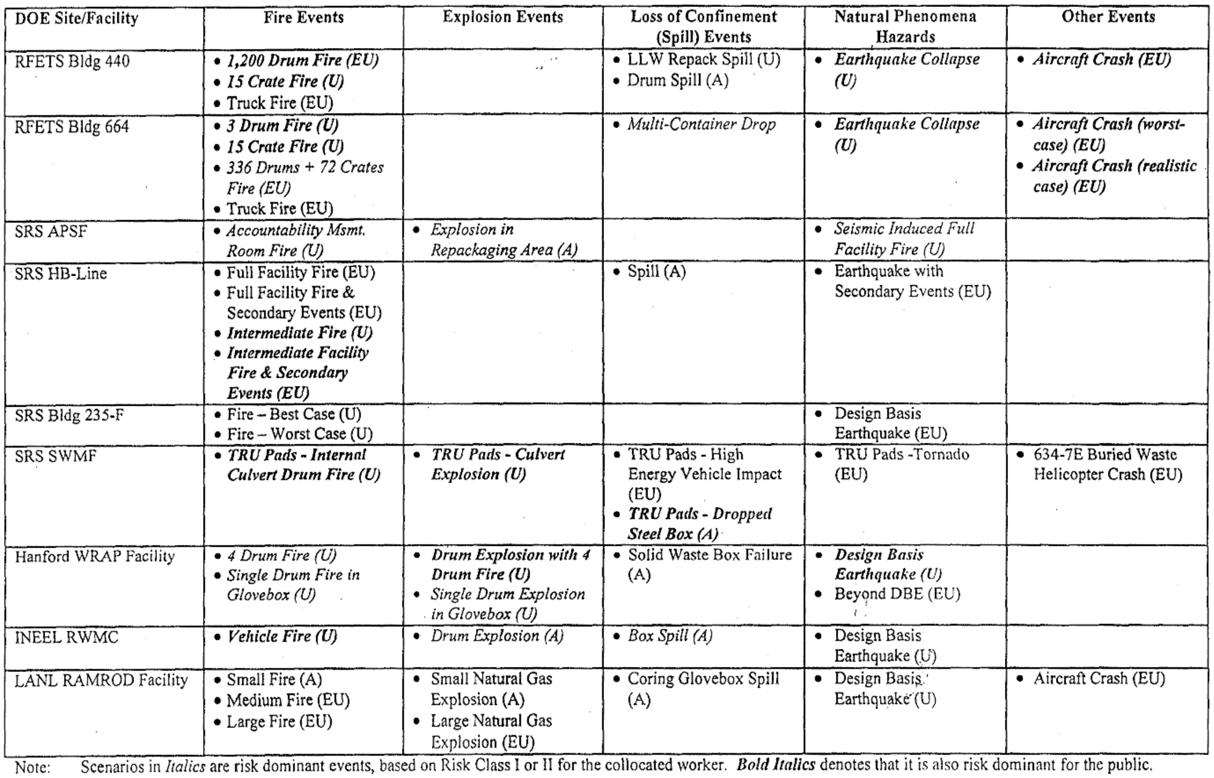
Water solubility tests of the chosen fixatives were carried out to determine the effect of prolonged exposure to water on applied fixatives. To do this, coupons were made by applying a fixative to a metal substrate and allowing it to fully cure. The fixatives were then removed from the metal substrate and cut into portions to fit into the test vials. Each vial was then filled with 5 mL of deionized (DI) water and stirred for 24 hours and visual inspection of the fixative was carried out after stirring. Each fixative tested is shown below with observational notes for the tested fixatives in Table 4-6.

Table 4-6. Observational notes after 24 hours of water exposure.

Fixative photos	Observation notes
	Product A: • Color of Product A turned from a clear yellowish to an opaque white. • Coupon had expanded its size and became swollen. • Surface was no longer tacky, and the exposed portion of the coupon was soft and flexible. • Water it was left in was also cloudy, showing some of the material had dissolved into the water.
	Product B: • Retained physical properties after exposure to water. • Sample floated on top of the water when exposed to the vial and had sunk to the bottom after 24 hours of exposure. • The material exhibited a taffy-like texture throughout the tests.
	Product C: • Displayed visible pores and striations within the cutout. • While the coupon was visible different, it still maintained the same basic properties of the control coupon including thickness and tackiness.
	Product D: • Immediately formed bubbles over the surface of the sample. • After two hours, fixative had completely dissolved in the water, resulting in a blue color and no solids.

5.0 Phase II: Proof-of-Concept for Intumescent Coatings Technologies and Down Selection

The findings from Phase I above highlighted the vulnerability of commonly-used fixatives to extreme thermal and environmental stressors associated with decommissioning activities, and particularly those postulated in accident scenarios in BIOs and safety basis documents across the DOE EM Complex (Figure 5-1). Furthermore, reports from the Defense Nuclear Facilities Safety Board (DNFSB) reinforce that the potential for a release of radioactive contaminants is especially acute during instances of fire, and these factors combined to prompt DOE EM to seek innovative technological solutions to enhance the fire resiliency of fixative technologies.



DOE Site/Facility	Fire Events	Explosion Events	Loss of Confinement (Spill) Events	Natural Phenomena Hazards	Other Events
RFETS Bldg 440	• ***1,200 Drum Fire (EU)*** • ***15 Crate Fire (U)*** • Truck Fire (EU)		• LLW Repack Spill (U) • Drum Spill (A)	• ***Earthquake Collapse (U)***	• ***Aircraft Crash (EU)***
RFETS Bldg 664	• ***3 Drum Fire (U)*** • ***15 Crate Fire (U)*** • *336 Drums + 72 Crates Fire (EU)* • Truck Fire (EU)		• *Multi-Container Drop*	• ***Earthquake Collapse (U)***	• ***Aircraft Crash (worst-case) (EU)*** • ***Aircraft Crash (realistic case) (EU)***
SRS APSF	• *Accountability Msmt. Room Fire (U)*	• *Explosion in Repackaging Area (A)*		• *Seismic Induced Full Facility Fire (U)*	
SRS HB-Line	• Full Facility Fire (EU) • Full Facility Fire & Secondary Events (EU) • ***Intermediate Fire (U)*** • ***Intermediate Facility Fire & Secondary Events (EU)***		• Spill (A)	• Earthquake with Secondary Events (EU)	
SRS Bldg 235-F	• Fire – Best Case (U) • Fire – Worst Case (U)			• Design Basis Earthquake (EU)	
SRS SWMF	• ***TRU Pads - Internal Culvert Drum Fire (U)***	• ***TRU Pads - Culvert Explosion (U)***	• TRU Pads - High Energy Vehicle Impact (EU) • ***TRU Pads - Dropped Steel Box (A)***	• TRU Pads -Tornado (EU)	• 634-7E Buried Waste Helicopter Crash (EU)
Hanford WRAP Facility	• *4 Drum Fire (U)* • *Single Drum Fire in Glovebox (U)*	• ***Drum Explosion with 4 Drum Fire (U)*** • *Single Drum Explosion in Glovebox (U)*	• Solid Waste Box Failure (A)	• ***Design Basis Earthquake (U)*** • Beyond DBE (EU)	
INEEL RWMC	• ***Vehicle Fire (U)***	• *Drum Explosion (A)*	• *Box Spill (A)*	• Design Basis Earthquake (U)	
LANL RAMROD Facility	• Small Fire (A) • Medium Fire (EU) • Large Fire (EU)	• Small Natural Gas Explosion (A) • Large Natural Gas Explosion (EU)	• Coring Glovebox Spill (A)	• Design Basis Earthquake (U)	• Aircraft Crash (EU)

Note: Scenarios in *Italics* are risk dominant events, based on Risk Class I or II for the collocated worker. ***Bold Italics*** denotes that it is also risk dominant for the public.

Figure 5-1. Sample of Accident Types Identified by DOE EM Complex.[18]

Having firsthand knowledge and experience in the use of intumescent coating technologies as a passive means to harden facilities and improve fire protection while serving in the U.S. military, FIU and SRNL researchers hypothesized that these technologies could be adapted to enhance the fire resiliency of current fixatives through a layering process. Although designed and intended to protect the structural integrity of buildings during fire (Figure 5-2), the potential new/repurposed use of this commercial-off-the-shelf (COTS) technology as an "incombustible fixative", capable of immobilizing residual radioactive contamination even when exposed to extreme thermal stressors, seemed viable.

Intumescent coatings swell between 50 to 100 times their original thickness into a robust, insulating char / foam upon exposure to heat, protecting the underlying material from fire by providing a physical barrier to heat and mass transfer. Flame spread is also inhibited through mechanisms common to other charring materials. The closed foam / char structure that forms inhibits the transport of volatiles to the environment and the transport of oxygen to unburned regions beneath the char, and the retention of mass in the char limits further involvement of the underlying materials in the fire. Furthermore, many intumescent coatings are exceptionally cost effective and can be easily applied via brush, roller, or sprayer to a wide variety of substrates (stainless steel, wood, sheetrock, sheet metal, etc.). Lastly, depending on the substrate and specific requirement, as little as two coats (10-30 mils) meet or exceed fire protection ratings for the construction industry as measured by ASTM E119 and ASTM E84. To put this in context, the ASTM E119 test subjects a given wall / structure to 24 gas flames that reach temperatures between 1800-2000° F for periods between 1-2 hours.

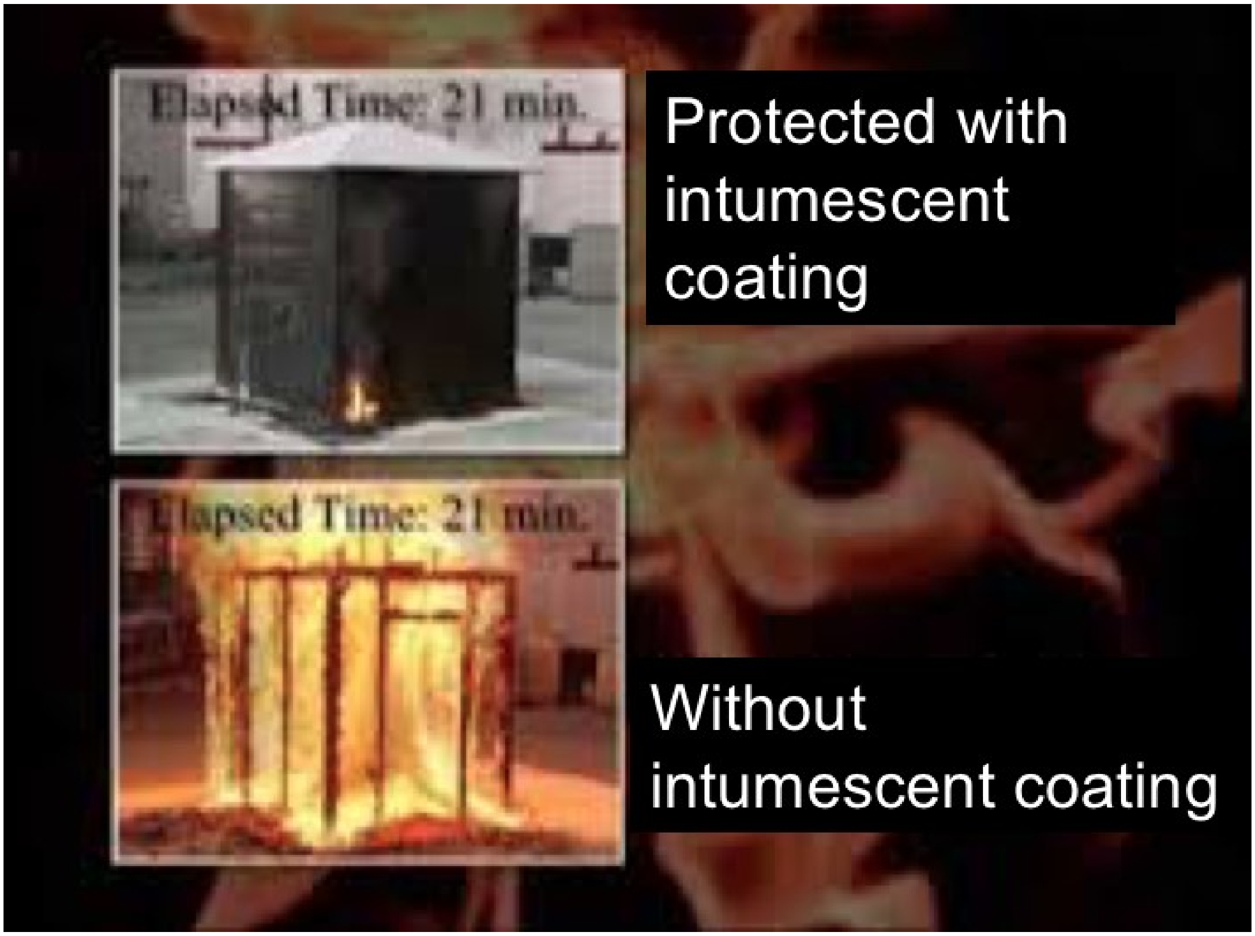


Figure 5-2. Fire protection from an intumescent coating technology.

With this as context and background, FIU ARC and SRNL moved forward and developed a series of proof of concept tests and evaluations on the potential of adapting COTS intumescent coatings to enhance the fire-retardant qualities of fixatives. Two approaches were ultimately investigated: 1) to ascertain whether the operational performance of current, commonly-used fixatives could be improved by layering them with an intumescent coating; and 2) could a down-selected intumescent coating serve as a stand-alone fixative in and of itself.

5.1 Selected Intumescent Coating Technologies[19]

The following intumescent coatings were evaluated during the proof-of-concept experiments:

1. Product F is an intumescent fire retardant and fire-resistant coating that is advertised to withstand extreme temperatures (up to 2000° Fahrenheit) for an extended period (over two hours). It provides a fire barrier to a wide variety of materials including sheetrock, wood, plaster, concrete, sheet metal, tin, foam, foam composite panels as well as advanced materials such as fiberglass and carbon fiber. Water-based and nontoxic, this intumescent coating is applied just like standard paints to a similar thickness. It has received the highest fire retardant ratings as measured by UL 263 / UL 723 / ASTM E-119 / ASTM E-84 / ASTM E-2768 / UL 10B / NFPA: 251 / NFPA: 255 / NFPA: 703 / NFPA: 252 / ULC S101 / ULC S102 / UBC 8.1 / UL 1715 / UBC 26-3 / UBC 7-1.

2. Product G is a water-based, non-toxic, thin film intumescent fire retardant and resistant coating fully tested to provide fire ratings required by the International Building Code for wall assemblies,

floor/ceilings assemblies, roof ceiling assemblies and individual structural members. It has been fire tested to multiple standards worldwide including ASTM E119, FM 4975, ASTM E662-97, ASTM E3675-98, ASTM E84, NFPA 286, and a host of others.

3. Product H is a sprayable, water-based material that dries to form a durable, elastomeric firestop coating. This material, when used as part of an assembly, will firestop building joints, perimeter joints (curtain wall), and through penetration seals. It is advertised to provide up to 4-hour fire protection in construction joints. It has been independently evaluated and rated against thermal stressors as tested to ASTM E1966 / UL 2079, ASTM E814 / UL 1479 / ULC-S115, and ASTM E84.

4. Product I is a single component, borate-free, high solids, low volatile organic compound (VOC) intumescent coating designed to provide fire protection to structural steelwork and has been independently tested at accredited laboratories to ASTM E119 / UL 263.

5.2 Proof-of-Concept Testing for Layering Concept of Selected Intumescent Coating Technologies[16]

In conducting proof-of-concept experiments to determine if layering commonly-used fixatives with COTS intumescent coatings could improve operational performance when exposed to thermal stressors, the basic testing protocols developed by FIU and SRNL outlined in Section 4.2 above were deemed appropriate. For the open flame series of tests, the exact same procedures were followed, and once the designated fixative was applied and cured on the 12 in x 12 in (30.48 cm x 30.48 cm) red oak substrate, it was then layered with the designated intumescent coating to the thickness suggested by the manufacturer and allowed to complete cure. The fixative-plus-intumescent-coating test coupons were placed into the open flame apparatus at the same 4 in (10.16 cm) distances from the propane torches to the center of the coupon. They were then subjected to a direct flame for 30 minutes (NOTE: the extended time period, as compared to the fixative-only test coupons at 5 minutes, was pursued because it became immediately apparent that the fixatives-layered-with-intumescent-coating test coupons were significantly more fire retardant and did not combust).

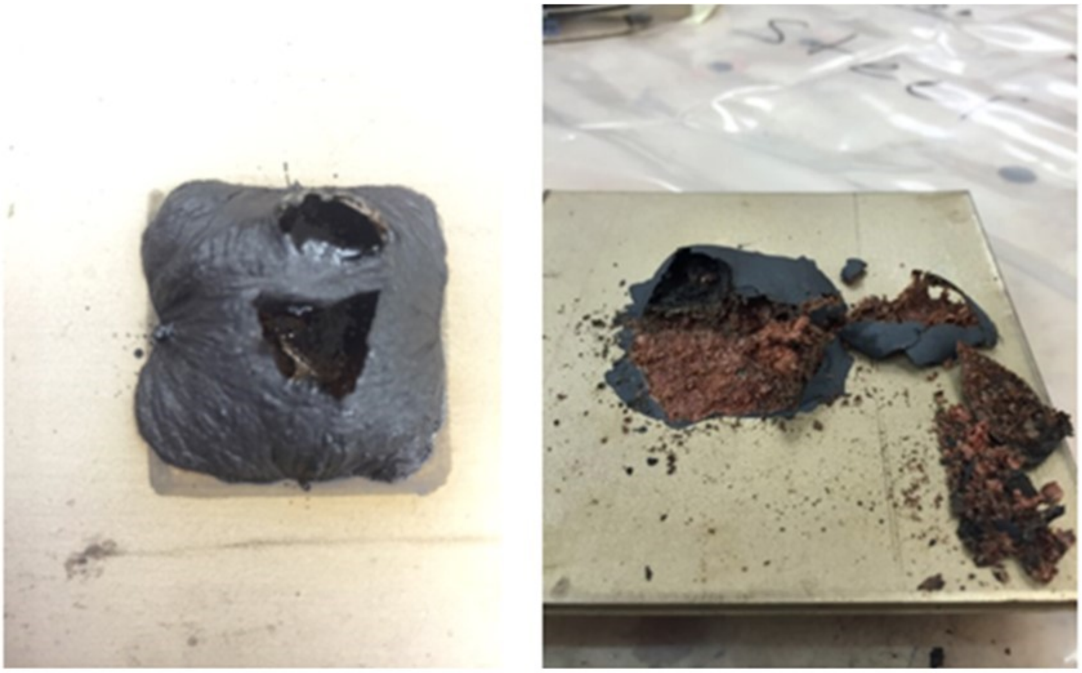
All 5 commonly used fixatives, when layered with the intumescent coating, displayed enhanced fire resiliency during the propane torch / open flame testing. In each instance, when compared to the results of the fixative alone, the fixative-plus-intumescent coating yielded better results in thermal insulation, minimization of smoke and flame spread, and noticeably reduced degradation to the fixative and substrate. As shown in Figure 5-3, the middle panel demonstrates the effectiveness of layering a fixative with an intumescent coating. The left and right panels are covered in fixative-only and burned completely off within 5 minutes of exposure to the flame source. The middle panel had a small area layered with Product F over Product A. Charring from the intumescent coating top layer immediately occurred, and protected both the substrate and the majority of the fixative lower layer for over 30 minutes (Figure 5-4). Flame spread / propagation was minimal, and never extended more than 2-3 inches from the center.

Figure 5-3. Demonstration of protection against direct flame when layered with intumescent coating.

Figure 5-4. Fixative underneath protective intumescent layer was relatively intact.

Although these proof-of-concept results were promising and demonstrated the potential for intumescent coating technologies to improve the operational performance of commonly-used fixatives across the DOE EM complex, safety basis personnel expressed some concern related to the charring process associated with the intumescent technologies tested. Specifically, the charring residue was quite frail and could be easily removed by blowing, brushing, air circulation, or water. The SRS 235-F PuFF Facility Risk Reduction team expressed a desire to have a fire retardant fixative material that would harden on a stainless steel substrate much like a ceramic and serve as a stand-alone fixative independent of any layering / combining of separate materials. This would operationally reduce application time by at least half, as only a single coat would nominally have to be applied without any interfacial concerns related to intumescent coating on fixative compatibility.

Of the intumescent coatings tested during the proof-of-concept, Product H demonstrated these characteristics. During a muffle furnace test on a stainless steel substrate, Product H hardened onto the surface and required a chisel to break it up and remove from the coupon (Figure 5-5). This led to the elimination of Products F, G, and I from further testing, and researchers focused benchmarking efforts on Product H for further testing of the product in other functional areas.



Product E – Significant discoloration, continued expansion, off gassing, mass loss, desiccation, cracking, and embrittlement (315.56 °C)

Product H – Baked and hardened (315.56 °C)

Figure 5-5. Results of Intumescent Coating Hardening After Muffle Furnace Testing.

5.3 <u>Headspace Analysis for Product H Intumescent Coating (IC)</u>[20]

Static headspace analysis of a curing IC directly samples the volatile organic compounds (VOCs) in the gas phase of a sealed sample.[21] Gas chromatography-mass spectroscopy (GC-MS) separates the VOCs in the sample and characterizes each component. Retention times and reference mass spectra are utilized to identify the compounds. Headspace GC-MS is a valuable technique for the detection of VOCs and the ease of operation and sample preparation saves time and money. GC-MS analysis was carried out using an Agilent system with a 6890 GC and 5973N MS with a fused silica capillary column DB-5MS (30 m x 250 μm x 0.30 μm). The temperature program was an isothermal hold at 34 °C for 2 min and then the temperature was raised to 150 °C at 9 °C/min. The method for analyzing VOCs was adapted from Linder and Jones.[22-23]

Two experiments were conducted in which Product H was cured at room temperature and at 110 °C. In the first experiment, Product H (0.8 g, < 1/8 in (.3175 cm) thick) was placed in a 150 mL Wheaton bottle, closed with a septum, and crimp-sealed with an aluminum seal. The sample was cured at room temperature (~23 °C) for 24 hr. At different times during curing, a 250 μL aliquot from the sample headspace was collected using a gas-tight syringe and hand injected into the GC-MS. For quantitative VOC analysis, Product H samples were heated to induce complete vaporization. Approximately 150 mg of Product H was added to a 150 mL Wheaton bottle and equilibrated at 110 °C for 20 minutes. All the VOCs should completely evaporate into the headspace during the heating period because of the small sample size and large headspace volume. After heating, a 250 µL aliquot from the sample headspace was collected using a gas-tight syringe and hand injected into the GC-MS. As a control, a known amount of internal standard, tetrahydrofuran, was added to Product H in a vial and thoroughly mixed. Then ~150 mg of the mixture was added to a 150 mL Wheaton bottle and heated at 110 °C for 20 minutes, and the headspace was analyzed. Calibration standards of VOCs (acetone, tetrahydrofuran, toluene, and *tert*-butanol) were prepared by injecting 1, 2, and 5 µL of the compound in 150 mL Wheaton bottles and sealing the bottles. The samples were equilibrated at room temperature for 30 minutes wherein the solvents completely evaporated. Additional standards were prepared through dilution by injecting 1 mL of the prepared gas samples into 150 mL Wheaton bottles. The standards were then analyzed in the same manner as the samples. The concentrations of the standards in parts per million (ppm) were calculated using equation

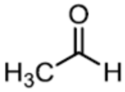
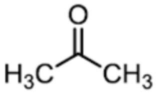
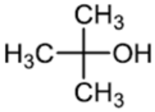
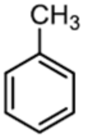
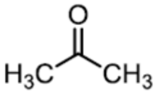
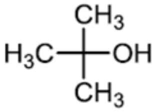
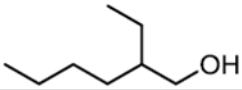
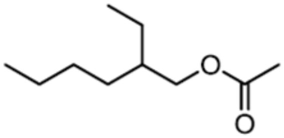
$$\boldsymbol{concentration\ (ppm)} = \left(\frac{\boldsymbol{RT}}{\boldsymbol{PM}}\right) \times \boldsymbol{N} \qquad (1)$$

where R is the ideal gas constant (62.4 L torr mol^{-1} K^{-1}), T is temperature (K), P is pressure (torr), M is molecular weight, and N is the concentration (mg m^{-3}). The measurements of VOCs using this method may be subject to bias from differences in partitioning of VOCs from the coating and headspace in samples relative to standards, and adsorption of VOCs to surfaces. The measurement bias from adding solid material to the Wheaton bottle is expected to be negligible as the sample is small relative to the headspace volume. Data were recorded in the SRNL electronic laboratory notebook system.

5.3.1 Product H Headspace Testing Results

Product H was cured at room temperature and analyzed by headspace GC-MS to detect the volatile compounds in the coating (Figure 5-6). The detected compounds are the solvents acetaldehyde, acetone, tert-butanol and toluene and these identified solvents are expected to evaporate from Product H during curing. Water was not detected in the GC-MS using the current method and experimental conditions. Table 5-1 summarizes the GC peaks' retention time, the identification of the isolated volatile compounds using MS, and the percent quality match with reference mass spectra. In addition, the identification of acetone, tert-butanol and toluene were confirmed using the standards' retention times. Product H samples were heated at 110 °C to quantitate the concentration of volatile compounds in the coating and the maximum expected amount of VOCs that will evaporate during curing. The headspace analysis of the heated Product H samples identified two new compounds, 2-ethylhexanol and 2-ethylhexyl acetate, which are commonly used solvents in coatings (Figure 5-7). Their high boiling points, >180 °C, and low volatilities are why the solvents were not detected in the room temperature headspace, and consequently, they are not expected to evaporate in significant amounts from the coating during room temperature cure.

Table 5-1. Results from headspace GC-MS analysis of Product H samples.

Sample	Retention time (min)	Compound	MS quality match
Product H (at room temperature)	1.61	Acetaldehyde 	78%
	1.82	Acetone 	83%
	1.94	*Tert*-butanol 	83%
	4.72	Toluene 	94%
Product H (at 110 °C)	1.83	Acetone 	78%
	1.94	*Tert*-butanol 	78%
	7.12, 7.20	No matches with MS library	N/A
	9.86	2-Ethyl hexanol 	86%
	11.98	2-Ethylhexyl acetate 	80%

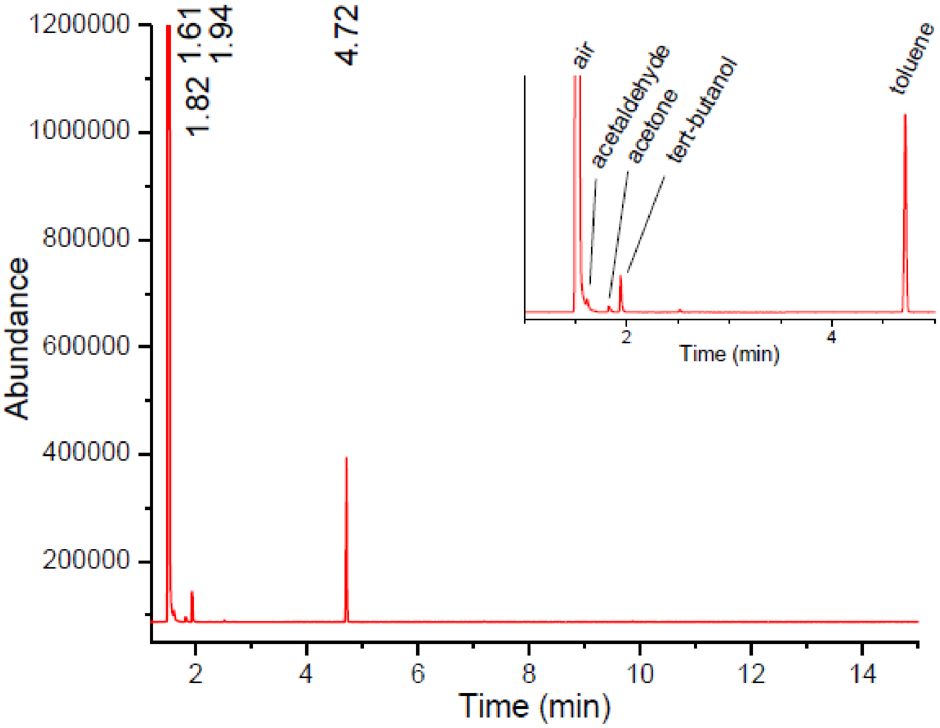


Figure 5-6. Gas chromatogram of the headspace of Product H in a sealed Wheaton bottle at room temperature. Inset: close up of chromatogram.

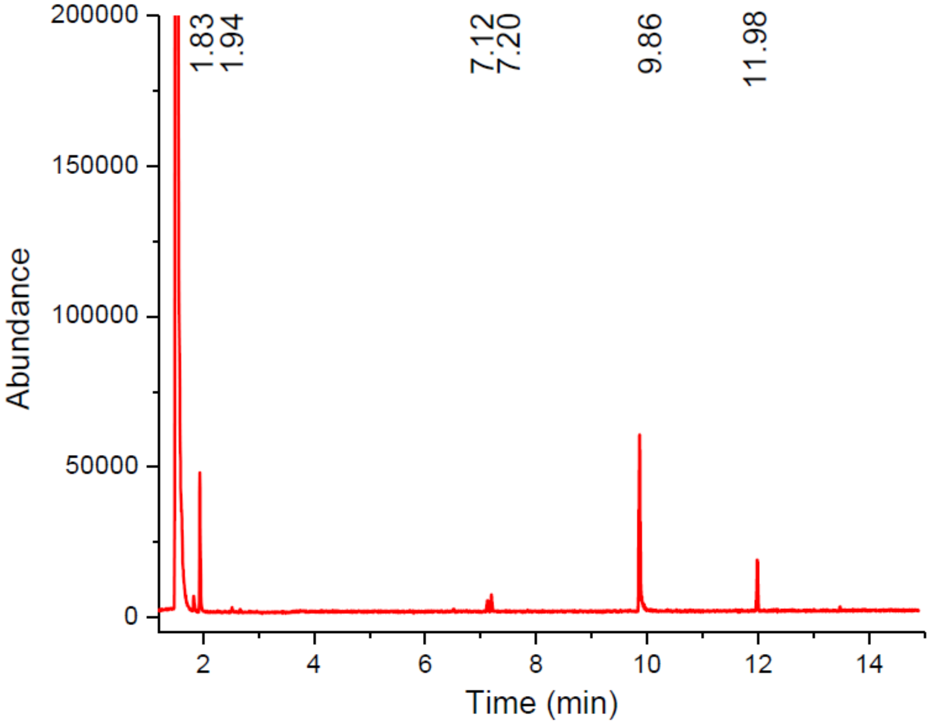


Figure 5-7. Gas chromatogram of the headspace of Product H in a sealed Wheaton bottle heated at 110 °C. Identified compounds are listed in Table 2.

The U.S. Occupational Safety and Health Administration (OSHA) requires safety data sheets (SDS) to list the hazardous chemicals in a product in quantities of 1% or greater.[24] As all the identified solvents in Table 5-1 are a flammable or combustible liquid, they should be listed in the SDS if they exceed 1% composition in Product H. As they were not listed, then these solvents must be in minute (<1%) quantities in Product H. The reported non-volatile content of Product H is 65%, which means that up to 35% of the volatile content could evaporate during curing. The average mass loss of Product H after curing for >24 hours for five samples was 31%. The majority of the mass loss is expected to be due to the evaporation of water. This assessment was supported by observation of condensation at the top of the sealed Wheaton bottle of the cured Product H sample. As explained below, the amount of VOCs in Product H is not enough to form visible droplets on the bottle.

The concentration of VOCs was determined by comparing the area of defined GC peaks for the heated Product H sample to standards. Note that several identified solvents were not quantitatively analyzed. First, the peak for acetaldehyde was not separated well enough from the air peak to be integrated. The peak for toluene was not observed; it is unclear why the presence of toluene in headspace analysis of Product H is not consistent. There were no standards for 2-ethylhexanol and 2-ethylhexyl acetate for this study. Assuming that all the volatile organic compounds went into the headspace of the Wheaton bottle, then the calculated concentration of acetone and tert-butanol in the headspace is 12 ppm and 43 ppm, respectively. This corresponds to acetone and tert-butanol being 0.003 and 0.01 weight percent of Product H, respectively. The weight percent of the other solvents are similar or within an order of magnitude based upon the area of the GC peaks. An internal standard of 1 wt. % tetrahydrofuran (THF) was added to Product H to verify that the total VOC content in Product H is less than 1%. Figure 5-8 shows the gas chromatogram of the headspace of Product H /THF mixture. The area of THF peak is much bigger (3 orders of magnitude) than the other volatile organic compounds. From these experiments, it was determined that the vast majority of the mass loss from curing of Product H is water, and Product H contains less than 1 wt. % of volatile organic compounds.

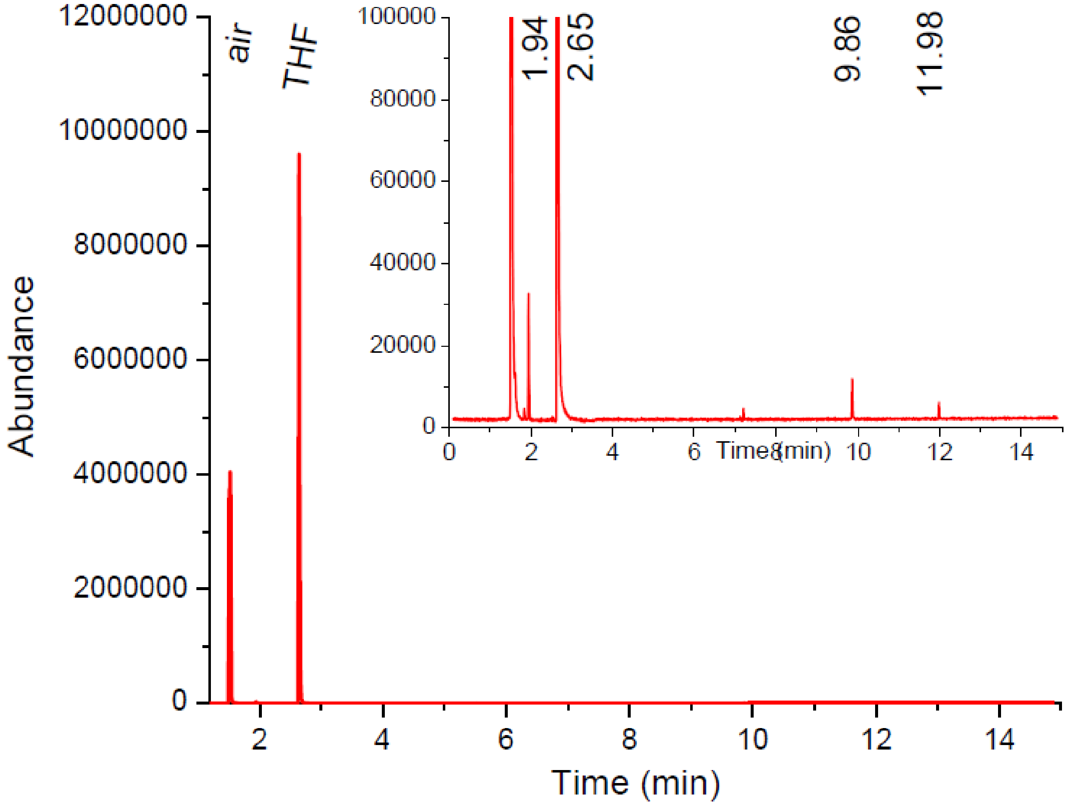


Figure 5-8. Gas chromatogram of the headspace of Product H with THF in a sealed Wheaton bottle heated at 110 °C. Inset: close up of chromatogram.

5.4 Environmental Impacts on Product H Intumescent Coating

To assess the longer-term environmental impacts on Product H, FIU monitored a set of control coupons over a 2-year period for comparison to the results from the anticipated SRS 235-F PuFF Facility hot demonstration. Control coupon 1 was maintained at FIU ARC's indoor laboratory under ideal conditions at ~72 ºF (22.22 ºC) and 30% RH. Control coupon 2 was maintained outside in ARC's hot cell testbed in Miami's ambient high temperatures and humidity. Weather conditions were monitored, and a monthly average temperature and humidity documented. Monitoring data collected by FIU was being evaluated for changes to the baseline data collected in December 2018. If a monthly measurement fell within the min-max range established, then that provided evidence that there had been no significant degradation of the coating or change in thickness due to environmental conditions. If a monthly reading fell outside of the min-max range, some amount of degradation or change in thickness had occurred.

5.4.1 Environmental Impacts on Product H Intumescent Coating Procedures

A step-by-step process was conducted with the manufacturer of the DeFelsko Positector 6000 FNTS probe, so there is a high level of confidence in the initial data points using the instrumentation (Table 5-2). The procedure is as follows:

1. The instrument was calibrated per the manufacturer's instructions and confirmed its accuracy on the DeFelsko 208.1 mils / 5.285 mm thickness standard coupon that came with the kit. Per the Certificate of Calibration (blue paper), FIU used the >2.5 mm +/- (0.01 + 3% of reading) formula, which means for the known thickness coupon our tolerance was +/- 0.169 (5.285*.03 + .01 = 0.169),

so our range was between 5.116 (min) to 5.454 (max). Then ten (10) measurements were conducted and all fell within the range, confirming accuracy of the instrument within the tolerance levels.

2. At each of the thirteen (13) designated points on the 12 in x 12 in (30.48 cm x 30.48 cm) 304 stainless steel control coupons, three (3) measurements were taken and then calculated the average, thereby establishing the anchor reading (column 5). The tolerance (column 6) was calculated and then determined the ranges (columns 7 and 8).

Table 5-2. Example of Establishing Baseline Measurements

Point	Trial #1 (mm)	Trial #2 (mm)	Trial #3 (mm)	Average (mm)	Average Tolerance (+/-) (mm)	Min Average Tolerance Range (mm)	Max Average Tolerance Range (mm)
1	4.32	4.28	4.29	4.30	0.14	4.16	4.44
2	4.95	4.99	4.95	4.96	0.16	4.80	5.12

*Note: Tolerance ranges were only calculated for the baseline measurements. All subsequent monthly measurements were an average of three measurements and were compared to this established range.

3. Before each monthly measurement the calibration of the instrument was confirmed as outlined in #1 above, and then the procedure outlined in #2 was followed and added to the matrix / record.

4. If the monthly reading fell within the min-max range established in baseline, then it was assumed that no significant degradation / changes in thickness occurred. If the monthly readings fall outside of the min-max range, then it was assumed that degradation / changes in thickness occurred.

5.4.2 Environmental Impact on Product H Intumescent Coating Results

The coating was applied to the minimum required thickness to the manufacturer's recommendation using the sprayer method (Figure 5-9). Point 3 on Coupon 1 was omitted from the monthly measurements because of the uneven topography which caused inaccurate readings. Initial testing indicated that the intumescent coating, which fully cures within about 24 hours, takes more time to completely stabilize (~2-3 months). There was up to a 5% difference (about 0.2 mm) at varying points in coating thickness readings in the first two months after the coating was initially applied. Once the measurements had stabilized, that became the baseline readings for the duration of the testing.

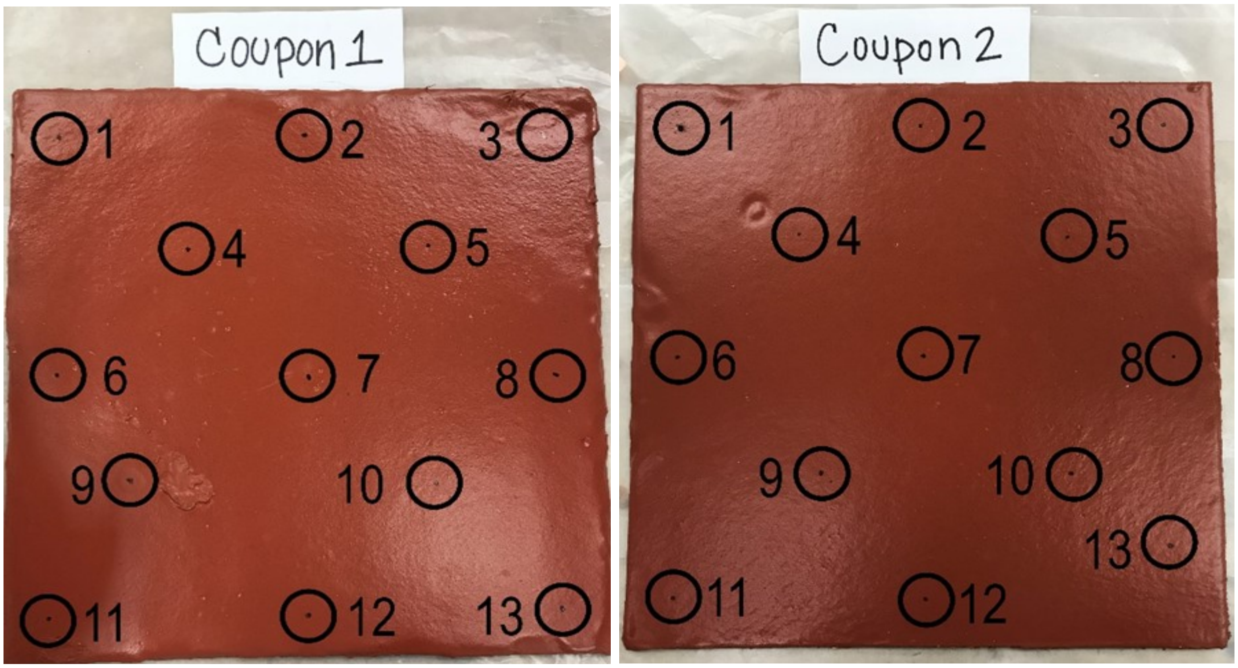


Figure 5-9. Coupon 1 (left) was maintained under ideal conditions and Coupon 2 (right) was exposed to varying environmental conditions.

The thickness measurements across all points on the 12 in x 12 in (30.48 cm x 30.48 cm) coupons remained within tolerances of the established baseline range, indicating that there has been no significant degradation in coating thickness to date. All the average monthly measurements for each point on the control coupons are shown in the tables below, with Table 5-3 and Table 5-4 taken for control coupon 1 which was kept in a controlled environment indoors and Table 5-5 and Table 5-6 corresponding to the thickness measurements taken for the coupon left outside.

Table 5-3. Control Coupon 1 (indoors) thickness test for first 7 months.

Point	**Baseline**		**Monthly Measurements 2019**						
	Min average tolerance (mm)	Max average tolerance (mm)	Jan.	Feb.	Mar.	Apr.	May	Jun.	Jul.
1	4.16	4.26	4.26	4.28	4.28	4.23	4.25	4.27	4.26
2	4.80	4.95	4.95	4.88	4.88	4.91	4.90	4.90	4.89
3	-	-	-	-	-	-	-	-	-
4	4.29	4.57	4.39	4.49	4.47	4.41	4.43	4.45	4.45
5	3.92	4.19	4.03	4.04	4.02	4.00	4.02	4.04	4.02
6	4.19	4.47	4.37	4.35	4.35	4.30	4.38	4.38	4.34
7	4.12	4.69	4.24	4.31	4.33	4.32	4.29	4.25	4.22
8	3.79	4.04	3.90	3.92	3.91	3.88	3.91	3.91	3.91
9	4.20	4.48	4.33	4.32	4.31	4.35	4.33	4.33	4.31
10	3.89	4.15	4.02	4.02	4.02	4.02	4.04	4.04	4.01
11	3.28	3.51	3.44	3.41	3.44	3.43	3.43	3.42	3.41
12	4.05	4.32	4.16	4.20	4.21	4.20	4.18	4.18	4.16
13	3.55	3.79	3.64	3.67	3.65	3.63	3.67	3.68	3.64

Table 5-4. Control Coupon 1 (indoors) thickness test for last 6 months.

Point	**Baseline**		**Monthly Measurements 2019**					**2020**		**Std Dev**
	Min average tolerance (mm)	Max average tolerance (mm)	Aug.	Sep.	Oct.	Nov.	Dec.	Jul.	Dec.	
1	4.16	4.26	4.26	4.24	4.24	4.25	4.24	4.33	4.34	0.032
2	4.80	4.95	4.95	4.88	4.89	4.90	4.89	4.85	4.97	0.033
3	-	-	-	-	-	-	-	-	-	-
4	4.29	4.57	4.42	4.37	4.46	4.46	4.44	4.44	4.48	0.033
5	3.92	4.19	4.03	4.06	4.03	4.00	4.01	4.03	4.08	0.021
6	4.19	4.47	4.38	4.40	4.36	4.32	4.32	4.42	4.41	0.036
7	4.12	4.69	4.21	4.25	4.24	4.26	4.21	4.34	4.39	0.055
8	3.79	4.04	3.90	3.89	3.92	3.92	3.91	3.97	3.90	0.021
9	4.20	4.48	4.36	4.28	4.39	4.35	4.38	4.33	4.25	0.037
10	3.89	4.15	4.01	3.98	4.04	4.01	4001	4.02	4.05	0.016
11	3.28	3.51	3.44	3.46	3.41	3.40	3.43	3.41	3.41	0.017
12	4.05	4.32	4.18	4.16	4.20	4.17	4.17	4.17	4.19	0.016
13	3.55	3.79	3.66	3.63	3.66	3.65	3.67	3.68	3.69	0.018

Table 5-5. Control Coupon 2 (outdoors) thickness test for first 7 months.

Point	**Baseline**		**Monthly Measurements 2019**						
	Min average tolerance (mm)	Max average tolerance (mm)	Jan.	Feb.	Mar.	Apr.	May	Jun.	Jul.
1	3.23	3.45	3.40	3.42	3.39	3.34	3.37	3.37	3.39
2	3.39	3.62	3.53	3.57	3.52	3.46	3.53	3.52	3.52
3	3.03	3.24	3.15	3.13	3.12	3.10	3.11	3.10	3.12
4	3.92	4.19	4.10	4.15	4.12	4.01	4.05	4.07	4.09
5	3.75	4.00	3.87	3.94	3.91	3.84	3.87	3.89	3.87
6	4.39	4.68	4.56	4.58	4.59	4.49	4.55	4.54	4.54
7	4.19	4.47	4.33	4.36	4.34	4.26	4.29	4.32	4.33
8	3.67	3.92	3.80	3.79	3.78	3.75	3.74	3.75	3.79
9	3.59	3.83	3.75	3.74	3.71	3.65	3.68	3.69	3.70
10	3.33	3.55	3.48	3.43	3.44	3.39	3.40	3.42	3.42
11	3.25	3.47	3.38	3.39	3.37	3.34	3.36	3.36	3.37
12	3.32	3.55	3.46	3.43	3.42	3.42	3.41	3.42	3.41
13	3.01	3.22	3.15	3.10	3.09	3.10	3.08	3.07	3.11
Average Temperature (high/low)			76° F 64° F	76° F 60° F	81° F 68° F	84° F 77° F	85° F 71° F	88° F 76° F	91° F 77° F
Average Daily Humidity			73%	75%	75%	74%	71%	68%	71%

Table 5-6. Control Coupon 2 (outdoors) thickness test for last 6 months.

Point	**Baseline**		**Monthly Measurements 2019**					**2020**		**Std Dev**
	Min average tolerance (mm)	Max average tolerance (mm)	Aug.	Sep.	Oct.	Nov.	Dec.	Jul.	Dec.	
1	3.23	3.45	3.38	3.38	3.37	3.38	3.37	3.38	3.43	0.023
2	3.39	3.62	3.51	3.49	3.51	3.52	3.51	3.53	3.55	0.025
3	3.03	3.24	3.12	3.10	3.10	3.13	3.11	3.10	3.22	0.031
4	3.92	4.19	4.07	4.06	4.08	4.10	4.08	4.09	4.09	0.032
5	3.75	4.00	3.87	3.85	3.89	3.90	3.89	3.90	3.95	0.031
6	4.39	4.68	4.52	4.54	4.54	4.57	4.52	4.60	4.56	0.030
7	4.19	4.47	4.33	4.30	4.31	4.33	4.30	4.37	4.34	0.028
8	3.67	3.92	3.76	3.77	3.75	3.76	3.74	3.76	3.74	0.019
9	3.59	3.83	3.72	3.67	3.69	3.72	3.70	3.69	3.71	0.026
10	3.33	3.55	3.43	3.44	3.41	3.42	3.41	3.40	3.42	0.022
11	3.25	3.47	3.37	3.32	3.36	3.38	3.38	3.34	3.35	0.019
12	3.32	3.55	3.41	3.41	3.43	3.42	3.44	3.43	3.41	0.016
13	3.01	3.22	3.12	3.11	3.12	3.12	3.15	3.06	3.07	0.028
Average Temperature (high/low)			92° F 78° F	92° F 78° F	91° F 79° F	89° F 78° F	83° F 69° F	93° F 79° F	77°F 62°F	
Average Daily Humidity			69%	74%	67%	72%	67%	72%	71%	

6.0 Phase III: Technology Application Methods, Cold Demo, and Full Rehearsals

SRNL and FIU researchers formally presented the data and results achieved to SRS 235-F site personnel, and with concurrence from DOE EM, the decision was made to proceed to Phase III of the research. Phase III focused on the following primary objectives: 1) develop and refine procedures for applying Product H inside the SRS 235-F PuFF Facility process cells given real-world constraints; 2) update the approved facility tooling list to accommodate any new technologies required for application of Product H ; and, 3) conduct crew training and a full rehearsal during a cold demo to inform formal test plan development and crew work procedures to support a future hot demo. The overall importance of this phase cannot be overstated, and it reinforces a common theme highlighted throughout this technical report. Specifically, a holistic technology development approach that addresses all facets related to the deployment of the technology itself must be pursued if a successful deployment is to occur. FIU ARC focused on the first two objectives and developed recommended procedures for application of Product H on both the horizontal (floor) and vertical (wall) surfaces of the process cells in their SRS 235-F Hot Cell Test Bed in Miami, Florida, and also identified recommended additions to the tooling list. SRNL and SRS subsequently confirmed those recommendations, and pressed forward with the execution of a cold demo and full rehearsal at their mock-up facility in Aiken, SC.

6.1 235-F Process Cell Test Bed in Miami, FL[19]

To support the accomplishment of the objectives outlined in Phase III, FIU ARC constructed a SRS 235-F Hot Cell Test Bed on site in Miami, FL. Extensive coordination on the design occurred among ARC, SRNL, and SRS 235-F site officials via site visits, emails, and phone conversations, and a final design was agreed upon by the respective stakeholders. SRS 235-F site personnel approved for release to FIU ARC the schematics for a typical cell scheduled for D&D at the facility (**Figure** 6-1), and based on projected funding and available space, ARC developed a to-scale, combined corner-middle cell configuration that mirrored the operating conditions (dimensions, glove ports, surface materials, obstacles/obstructions, etc.) encountered in cells 6-9 at the facility (**Figure** 6-1-Figure 6-4). This layout was determined as the most optimal configuration to ensure tooling could adequately be brought into the corner cell through the bag in port, retrieved and handled in gloves, and passed through the port between the cells. Where tooling failed to meet this pass-through requirement, other options had to be pursued as these constraints cannot be negotiated within the 235-F hot cells.

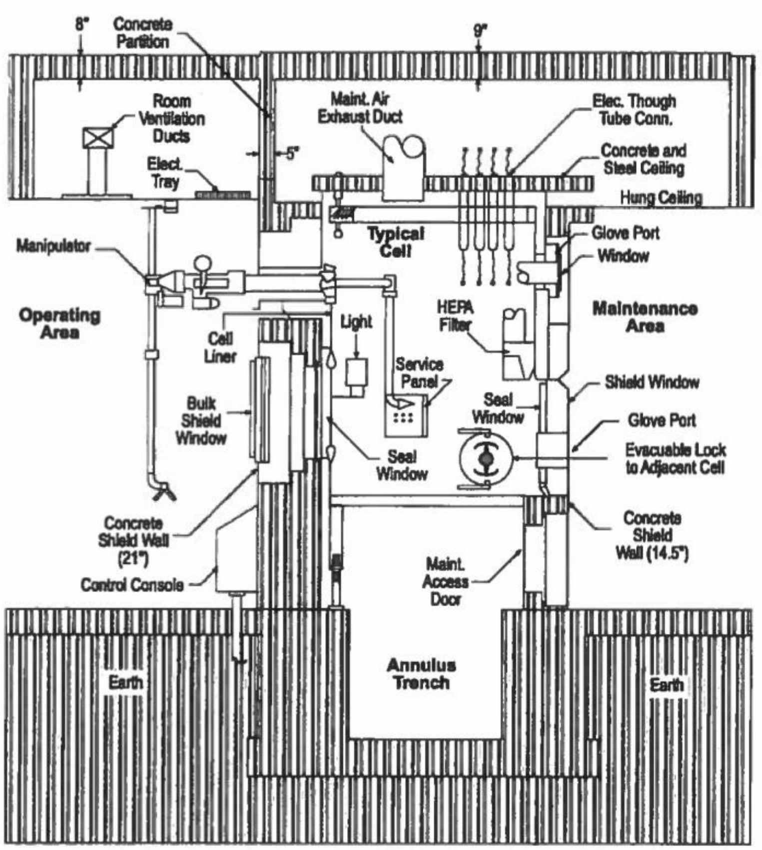


Figure 6-1. Typical processing cell at SRS 235-F PuFF Facility.

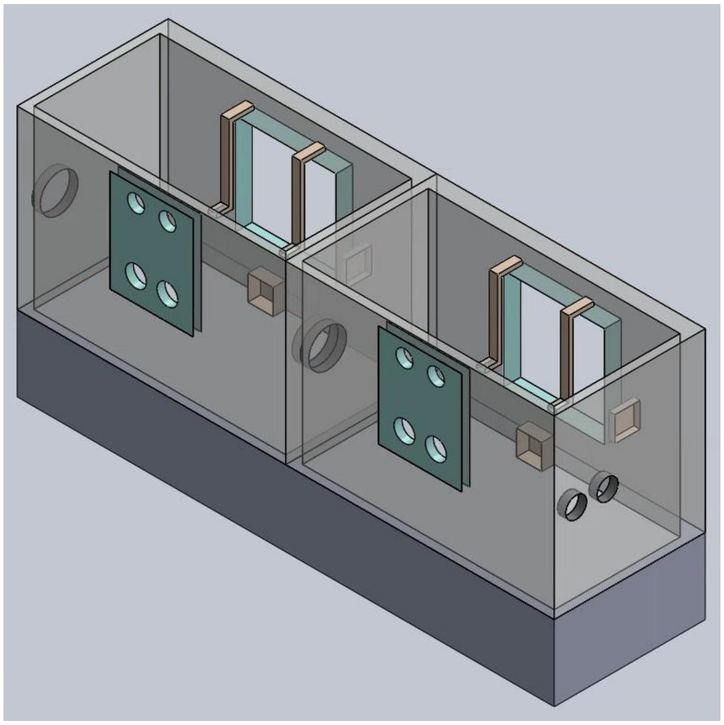
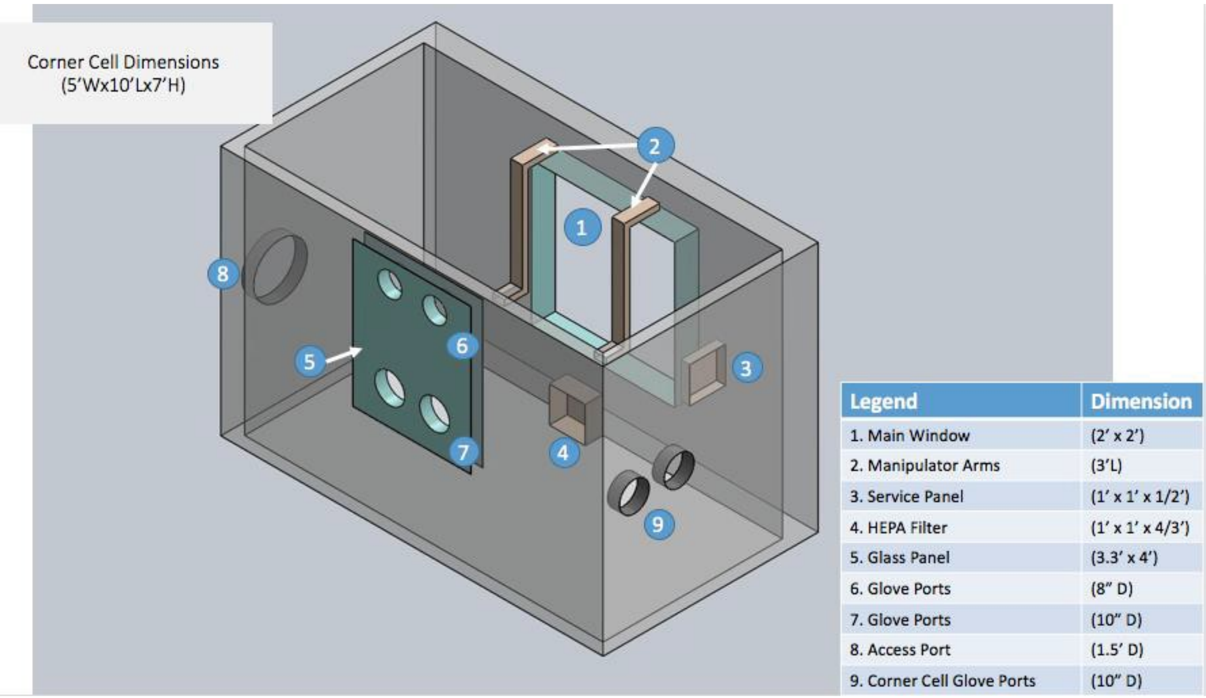
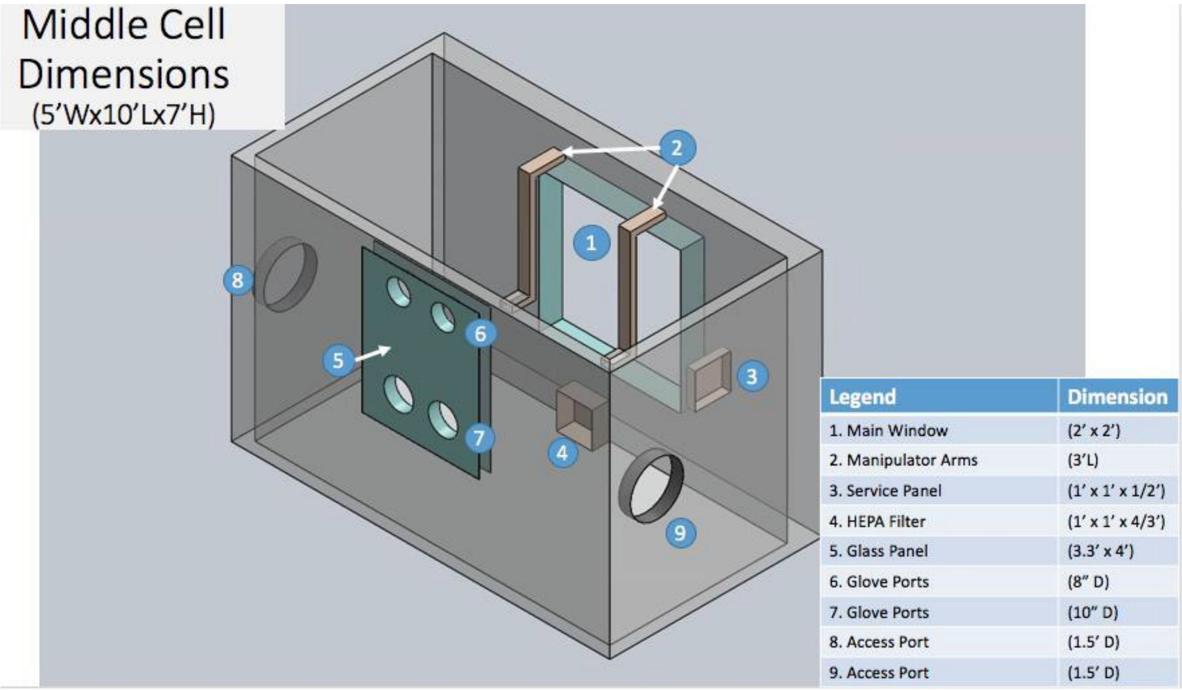


Figure 6-2. FIU ARC middle and corner cell configuration design.



Legend	Dimension
1. Main Window	(2' x 2')
2. Manipulator Arms	(3'L)
3. Service Panel	(1' x 1' x 1/2')
4. HEPA Filter	(1' x 1' x 4/3')
5. Glass Panel	(3.3' x 4')
6. Glove Ports	(8" D)
7. Glove Ports	(10" D)
8. Access Port	(1.5' D)
9. Corner Cell Glove Ports	(10" D)

Figure 6-3. FIU ARC corner cell dimensions.



Legend	Dimension
1. Main Window	(2' x 2')
2. Manipulator Arms	(3'L)
3. Service Panel	(1' x 1' x 1/2')
4. HEPA Filter	(1' x 1' x 4/3')
5. Glass Panel	(3.3' x 4')
6. Glove Ports	(8" D)
7. Glove Ports	(10" D)
8. Access Port	(1.5' D)
9. Access Port	(1.5' D)

Figure 6-4. FIU ARC middle cell dimensions.

Figure 6-5. Construction of SRS 235-F Hot Cell Test Bed at FIU ARC.

FIU completed the construction of the hot cell test bed at FIU ARC's Outdoor Technology Testing & Demonstration Facility in May 2017. The dimensions of each cell are 5 ft wide x 10 ft long x 7 ft high (1.52 m x 3.05 m x 2.13 m) and include a 3 ft (.91 m) raised floor. Pass-through ports and glove ports were sized and positioned in collaboration with SRNL/SRS to closely mimic the actual conditions at the facility. Sheets of 304-stainless steel were installed to represent the surfaces found in the hot cells. During May 2017, Mike Serrato, Aaron Washington and Connor Nicholson from SRNL as well as Jack Musall (SRS Project Engineer for the D&D of the hot cells at SRS 235-F) had the opportunity to perform a detailed review and tour of the constructed Hot Cell Test Bed at FIU ARC, and confirmed its suitability to achieve the objectives outlined in this phase (Figure 6-6).

Figure 6-6. Completed interior (left) and exterior (right) photos of the hot cell mockup constructed at FIU to represent SRS 235-F PuFF Process Cells 6 and 7.

6.2 Tooling Constraints - SRS 235-F Risk Reduction Tooling List[25]

In support of the deactivation project, the 235-F Deactivation Project Team identified various tools to remove and/or immobilize the residual radiological material within the PuFF Facility Process Cells. In order to ensure compliance with facility regulations and subsequent management of waste/material from intrusive activities, any tool used in the 235-F PuFF Facility Cells must be on the 235-F Risk Reduction Tooling List, Rev 0, dated 26 January 2015.[25] The tooling list was designed as a living document able to meet the Risk Reduction team's needs, and has undergone multiple revisions since initial publication. Figure 6-7 below is a direct extract from that document. It highlights the tools that were approved for use in the 235-F PuFF Facility Cells for the application of decontamination agents and fixatives. This document served as the initial starting point for FIU ARC as researchers tested various application methods for Product H. However, based on conversations with the SRS 235-F project manager and SRNL research scientists, the possibility to add tools to the approved tooling list, if there was an operational requirement to do so, was emphasized.

TOOL	APPROVED USAGE	PHOTO	REF. DRAWING OR PART NO.	COMBUSTIBLE COMPONENT SUBJECT TO REFERENCE 4	APPLICABLE FAI (per Reference 7)
Roller	Apply decontamination agents and fixatives.		SRNL Drawing No. R-R4-F-00153	Yes	SDD-2015-00002
EXTENSION COMPONENTS					
Extension, End	Use in combination with various tools (e.g., Extension Rake) and the Middle Extension. Allows the various tools to be used at distance.		SRNL Drawing No. R-R1-F-00145	Yes	SDD-2015-00002
Extension, Middle	To be used in combination with various tools (e.g., Extension Rake) and the End Extension. Allows various tools to be used at distance.		Fabricated by SRNS Construction or SRNL Drawing No. R-R1-F-00151	No	SDD-2015-00002
Paint Brush, various sizes (No wood, plastic handle only)	For brushing off items and components in the vicinity of the gloveports, and for transferring sweepings to a dust pan.		Various manufacturers and models	Yes	SDD-2015-00002

Figure 6-7. Approved tools for fixative application per the 235-F Risk Reduction Tooling List.

FIU ARC was encouraged to identify new tools and methods that may facilitate the application of the Product H given the constrained environment encountered in the SRS 235-F hot cells. Some general limiting factors highlighted by SRS site personnel were that any tools / application methods to support D&D activities need to be: 1) self-contained; 2) have no requirement for a tether extending outside the cell; 3) be able to be introduced through the bag-out port (~18 in (45.72 cm) diameter); and, 4) ultimately be approved by Industrial Safety, Industrial Hygiene, Radiological Protection, and F-area Engineering.

6.3 Development of Application Methods

Once construction of the SRS 235-F Hot Cell Test Bed was complete, FIU ARC began experimenting with various methods to efficiently apply the Product H. In addition to meeting the compliance criteria outlined above, special attention was also placed on developing procedures that: 1) mitigated worker fatigue; 2) improved cure times for the material; 3) negated the requirement for multiple coating applications and, by extension, requirements for dressing out the crew; 4) minimized the potential for disturbing the residual contamination and making it airborne; and, 5) maintained the fire rating integrity of the intumescent coating.

6.3.1 Horizontal Surfaces

With 95% of the assayed contamination residing on the floor of the PuFF Facility process cells, FIU ARC decided to focus on the horizontal surfaces, first and foremost. It was assessed that once application of the Product H was accomplished on the floor; the majority of the assayed residual contamination could be fixed/immobilized. Given the viscosity of Product H is 120,000 centipoise (CPS) and had the relative consistency of pancake batter, FIU ARC assessed that the material could be slowly poured and spread across the floor utilizing the already approved tools on the 235-F Risk Reduction Tooling List in a single application.

Researchers performed a small-scale test of slowly pouring Product H onto a 12 in x 12 in (30.48 cm x 30.48 cm) area within the hot cell test bed to the requisite thickness of 1/8 in (0.3175 cm) in a single pour. This slow-pour method showed significant promise in reducing worker time and had the added benefit of mitigating the potential for disturbing residual contamination during the application of the intumescent coating on horizontal surfaces in hot cells. Product H cured within 24-hours of application with a heat index of around 100 ºF (37.78 ºC). To confirm Product H maintained its fire rating when applied in this manner, FIU performed fire testing on the coupon. This testing yielded positive results with minimal to no flame propagation or smoke and demonstrated excellent thermal insulation provided by Product H (Figure 6-8).

Figure 6-8. Fire testing test coupon after slow-pour application.

Consequently, FIU ARC proceeded with a larger-scale test and evaluation of the technique and sectioned off a 5 ft x 5 ft (1.52 m x 1.52 m) area of the hot cell to further evaluate the effectiveness of using a simple slow-pour method of applying an intumescent coating to the floor of the hot cell test bed. The following tools were used (Figure 6-9):

- Container – 2.5 qt (2.37 L)-size plastic bucket to hold/transport the IC
- Gripper - used to maneuver a 2.5 qt (2.37 L)-size plastic container of IC
- Custom wooden T-shaped extension tool - used to spread the IC. To develop this tool, FIU ARC connected a 13 in (33.02 cm) wooden head to the approved extension handle on the SRS tooling list.

FIU ARC poured Product H into a 2.5 qt (2.37 liters) plastic container outside the hot cell, filling the container with 1 to 1.25 quarts (.95-1.2 L) of the coating material to keep the weight between 2 to 3 pounds to minimize worker fatigue. The container and tools were then passed through the bag in/bag out port. Maneuvering the 2.5 qt (2.37 liters) container to various locations within the hot cell by manipulating the gripper from the glove-ports was relatively easy and allowed for targeted pouring. After marking the hot cell border at 1/8 in (0.318 cm) above the floor, FIU poured the IC from the container at a height of about 1 to 3 in (2.54 to 7.62 cm) above the floor to minimize any potential for splatter or disturbance of any residual contamination that may be present in a radioactive environment. FIU then spread and smoothed the IC across the floor area using the wooden T-shaped tool, using the 1/8 in (0.318 cm) markings along the perimeter of the hot cell as a thickness guide. The custom wooden T-shaped extension tool allowed access to all locations within the hot cell test bed from the glove ports and easily reached the corners. An area of 5 ft x 5 ft (152.4 cm x 142.4 cm) was coated to 1/8 in (0.318 cm) thickness in approximately 15 minutes once the tools and Product H containers were in the hot cell.

Figure 6-9. Tools used for slow pour method to apply IC to hot cell floor.

Total curing time after the slow pour application was 48 hours under hot and humid weather conditions (temperatures 75°-97 °F and humidity 45%-93%). No international testing protocols for confirming curing of an intumescent coating at thicknesses of 1/8 in (0.318 cm) or greater in a radioactive environment were identified. To confirm curing, FIU developed a basic pressure test based on previous experiments with the material. Using a white paper towel, FIU pressed firmly on the test area, removed the paper towel, and observed the surface for any indentation and/or any discoloration of paper towel. For areas within the hot cell test bed that were beyond arms reach, this test was accomplished using a gripper tool. All horizontal surfaces were cured at 48 hours after the slow pour application method (Figure 6-10).

Thickness of the coating was confirmed using the same Defelsko PosiTector-6000 FNTS (0-250 mils, Ferrous + Non-Ferrous) described in Section 5.4.1 of this document. This instrument conforms to ISO 2178 / 2360 / 2808, ASTM B499 / D1186 / D1400 / D7091 / E376 / G12, BS3900-C5, SSPC-PA2 and others. After two applications using the slow-pour application method for horizontal surfaces previously described, all areas of the coated floor met the requisite 1/8 in (0.318 cm) thickness requirement for Product H's fire rating.

Figure 6-10. Steps for slow pour application to horizontal surfaces.

Based on discussions with SRS personnel to ease potential worker load, FIU ARC divided the hot cell floor application into two (2) segments using a 2 ft x 4 ft (0.61 m x 1.22 m) as the segregation material due to convenience and ready availability; however, other materials, such as stainless steel, could be used depending on the facility's requirements and preferences. The properties of Product H allowed it to cure along the seam between sections with no noticeable gaps in the seam. The relative ease and minimal time needed to apply the IC via this method may afford the end user the opportunity to forego dividing the area into sections.

The slow pour method for the floor/horizontal surfaces proved very effective during application, significantly reducing time and effort while greatly facilitating application to the requisite thickness level. With an estimated 95% of the contamination residing on the hot cell floors at the SRS 235-F facility and given the composition and characteristics of the IC being tested, the slow pour method proved to be a viable application method for horizontal surfaces that would expedite application and minimize disturbance of any residual contamination.

6.3.2 Vertical Surfaces

Although application of the IC using the site approved tools, specifically the extension pole and a roller brush, was achieved on the wall surfaces of the hot cell, it became readily apparent that achieving the requisite 1/8 in (125 mils or in 0.318 cm) thickness would be overly labor intensive and time consuming, requiring multiple applications as highlighted in Figure 6-11 below.

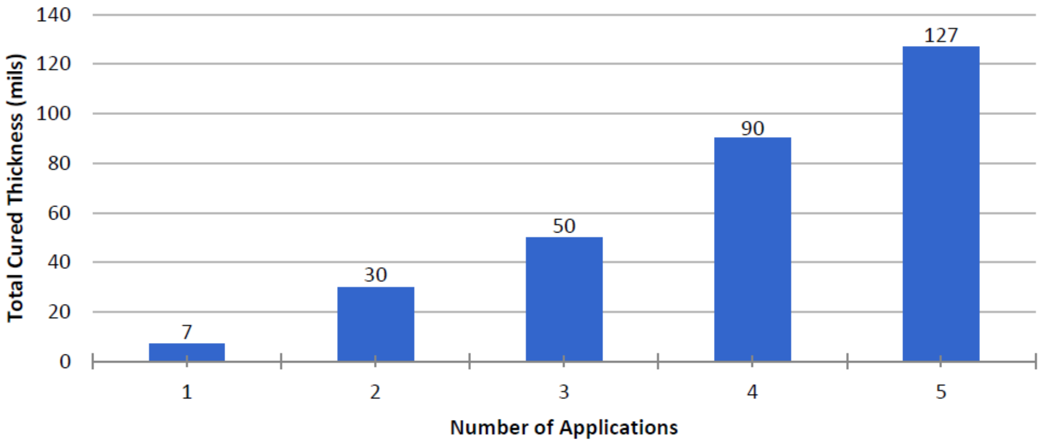


Figure 6-11. Average cured thickness achieved per application with roller (125 mil final target).

The data represents the average total cured coating thickness for five (5) stainless steel substrate coupons (12 in x 12 in (30.48 cm x 30.48 cm)) on which Product H was applied via roller. A period of 24 hours was allowed between each roller application to allow for curing, which extended the overall application period to approximately 120 hours in order to achieve the manufacturer specified requisite thickness of 1/8 in (.318 cm) to ensure the IC maintained its fire rating. Though a larger sample size would improve the statistical relevance, dialogue with SRNL and site personnel quickly determined that alternative application methods would need to be identified to be operationally feasible in a contaminated environment onsite for vertical surfaces such as walls.

6.3.2.1 Introduction of Handheld Sprayer for Application on Vertical Surfaces

A particular challenge anticipated with any COTS handheld sprayer was its compatibility with the down-selected IC technology. As stated above, the viscosity of Product H is 120,000 CPS. As context, the viscosity of water is 1 CPS and the viscosity of peanut butter is roughly 250,000 CPS. After a literature search and discussions with SRNL and SRS site personnel, the Graco Ultra Max cordless airless handheld paint sprayer (model number 17M367) was selected for initial testing.

GRACO TrueCoat Pro II Cordless Paint Sprayer: The TrueCoat® Pro II is a cordless airless sprayer with a ProControl™ Pressure Control System that allows the user to adjust the pressure to spray a wider variety of applications. It is providing a full motion of spraying with the new Tilt-N-Spray pendulum suction tube design, and is powered by a new, lighter weight G20 lithium-ion battery. Product dimensions are 10.75 in x 5 in (27.3 cm x 12.7 cm) and weighs 6.86 lbs (3.11 kg) (Figure 6-12).

Figure 6-12. GRACO TrueCoat Pro II cordless paint sprayer.

FIU ARC conducted a small-scale evaluation of the sprayer on a 20 in x 20 in (50.8 cm x 50.8 cm) vertical piece of stainless steel and was able to uniformly apply the selected IC technology using the handheld sprayer to the requisite 1/8 in (0.318 cm) in one application in less than a minute. A larger-scaled test was conducted using the corded version of the GRACO on a 5 ft x 7 ft (1.52 m x 2.13 m) area of wall located in the hot cell and yielded similar results. The area was coated in two applications over a 48-hour period and cured to the requisite 1/8 in (0.318 cm).

Figure 6-13. GRACO Ultra Max cordless airless handheld paint sprayer loaded with Product H.

A rough order of magnitude calculation was performed to determine the volume of Product H needed to coat all hot cell surfaces (floor, ceiling, 4 walls) to 1/8 in (0.318 cm) thickness.

Table 6-1. Required volume of Product H to coat interior of mock-up cold cell.

	Hot Cell Floor/ Ceiling	Hot Cell Side Walls	Hot Cell Back/ Front Walls
Surface Dimensions	5' x 10'	5' x 7'	10' x 7'
Coating Thickness	1/8"	1/8"	1/8"
Volume of Coating	900 in^3	630 in^3	1260 in^3
Volume of Product	3.9 gallons	2.8 gallons	5.5 gallons

The total volume of product needed to apply a 1/8 in (0.318 cm) thick coating to all of the surfaces would be approximately 24.4 gallons [3.9 gal x 2 (floor & ceiling) + 2.8 gal x 2 (side walls) + 5.5 gal x 2 (front/back walls)]. With the retail cost of the IC being approximately $425 for a 5-gallon container, the approximate cost to coat one hot cell of this size would be $2,125. These estimates do not include the effect of other hot cell structures such as the bag-in/bag-out and pass-through ports, windows, etc. In addition, the calculations do not consider potential product losses and other variables. The estimated volume of product should therefore include a safety factor multiplier to ensure that enough IC is available.

6.4 SRNL Cold Demo/Rehearsal[26]

SRNL Process – Mockup Spray Testing

Following the results of the FIU cold demonstration, lessons learned on application techniques were examined and a test plan was developed to outline objectives for a hot demonstration that needed to be benchmarked prior to facility entry in order to train operations personnel.[27] From the FIU results, it was found that spraying using the Graco Ultra brand sprayers was the best-in-class application method vs. traditional painting due to the time associated with coating: 1 qt (0.95 L) of Product H could be sprayed over the course of about a minute and a half with reloading time of cartridges averaging about two minutes whereas a roller method required significantly more physical effort and time to put equivalent material on the coated surface.

For hot cell tests and the actual application, the corded version of the GRACO Ultra was used. This decision was made because the batteries would be an added source of mixed waste. Waste Acceptance Criteria (WAC) compliance requires a minimization of preventable waste and the use of batteries was deemed counter to this mission as the corded version was able to provide the same functionality as the cordless version. Maintaining control of the spraying pattern was also found to be relatively easy with minimal overspray in the training environments. Likewise, for horizontal coating, the pour and spread method was found to be the best-in-class over spraying or rolling onto the surface. Due to the weight of the sprayer and the angles involved for the floor, the sprayer was disqualified as a means for coating. Traditional paint rolling also exhibited the same problems as with vertical surfaces: the amount of time and effort it took to coat the floor vs. the pouring method proved to be excessive.

From these lessons learned, SRNL and SRS personnel embarked on a cold demonstration in a prebuilt mockup that replicated Process Cell 7 in 235-F. Initial efforts were performed outside of the mockup to familiarize operators with the new tooling (sprayer, roller, thickness gauge) they would be using to perform these operations. All cold demonstration operations were applied to either paper or cardboard to keep the mockup enclosure as clean as possible. Coverings were also applied to all windows, where possible, to protect the windows from getting Product H on them. This was also performed to mimic operations to be performed in the hot cell, as the windows were to be maintained, so application of Product H to any windows was requested to be minimized. All activities were performed from the maintenance gloveports within the hot cell due to manipulators in the 235-F building being inoperable. A summary of these activities is shown in Figure 6-14. Areas that were scrutinized were: 1) how well operators could retrieve all tooling from the bag in port, 2) whether the coating method was able to achieve the requisite 1/8 in (0.318 cm) coating from the gloveports, 3) how well the associated tooling (sprayer, cartridges, spreader, thickness gauge) could be manipulated from the gloveports, and 4) identifying any additional obstacles that came from working within the enclosed space through gloves.

Figure 6-14. a) Vertical surface sprayed outside of cell mockup to familiarize operators with sprayer setup, functions, and cleaning. b) Vertical surface sprayed inside SRS cell mockup that is of the same geometry of the cell to be sprayed (235-F Process Cell 7). c) Horizontal pouring/spreading of the IC on the floor of the mockup. d) Material thickness measurement verification.

The cold demonstration proved successful and the hands-on training the operators received through this process was valuable as the team moved towards the hot demonstration. It was noted that adhesion to the cardboard and paper vs. stainless steel was significantly different and lead to sagging when too much material was applied. This resulted in one instance in a layer of Product H sloughing completely off the substrate. This led the SRS team to modify application to two separate coatings for each surface and gave operators a sense of when "too much" had been applied that would likely result in significant sloughing/sagging/running. It was also found that if the material was applied in a stagnant environment, it took significantly longer to dry as compared to a system where active ventilation was circulating air. This was induced within the mockup through a simulated air venting system and proved to cure the material in approximately 48 hours once ventilation was turned on as opposed to the material still being completely uncured after 24 hours with no induced ventilation. Another take away from this experience was to ensure a metal backing was used to test the thickness of the applied material to paper. Previous measurements without a metal backing gave artificially low results due to the physics of the detector's measurement, and once the sample was placed on a piece of stainless steel, sufficiently thick measurements were obtained. In practice, this would not have been an issue as all surfaces Product H was to be applied to would be stainless steel.

7.0 Phase IV: Hot Testing in 235-F[27]

Taking the lessons learned from the SRNL/SRS cold demonstration, two application sites were identified within building 235-F, and a work package summarizing activity in these areas was prepared (Appendix A):

1. Process Cell 7 which had already undergone decontamination efforts and was known to contain approximately 1g of Pu-238 holdup,[28] and
2. the Entry Hood leading into a glovebox train attached to Process Cell 1 that had not been decontaminated at all and was known to contain approximately 0.33g of Pu-238.[28] Pictures of these two areas prior to application are shown in Figure 7-1.

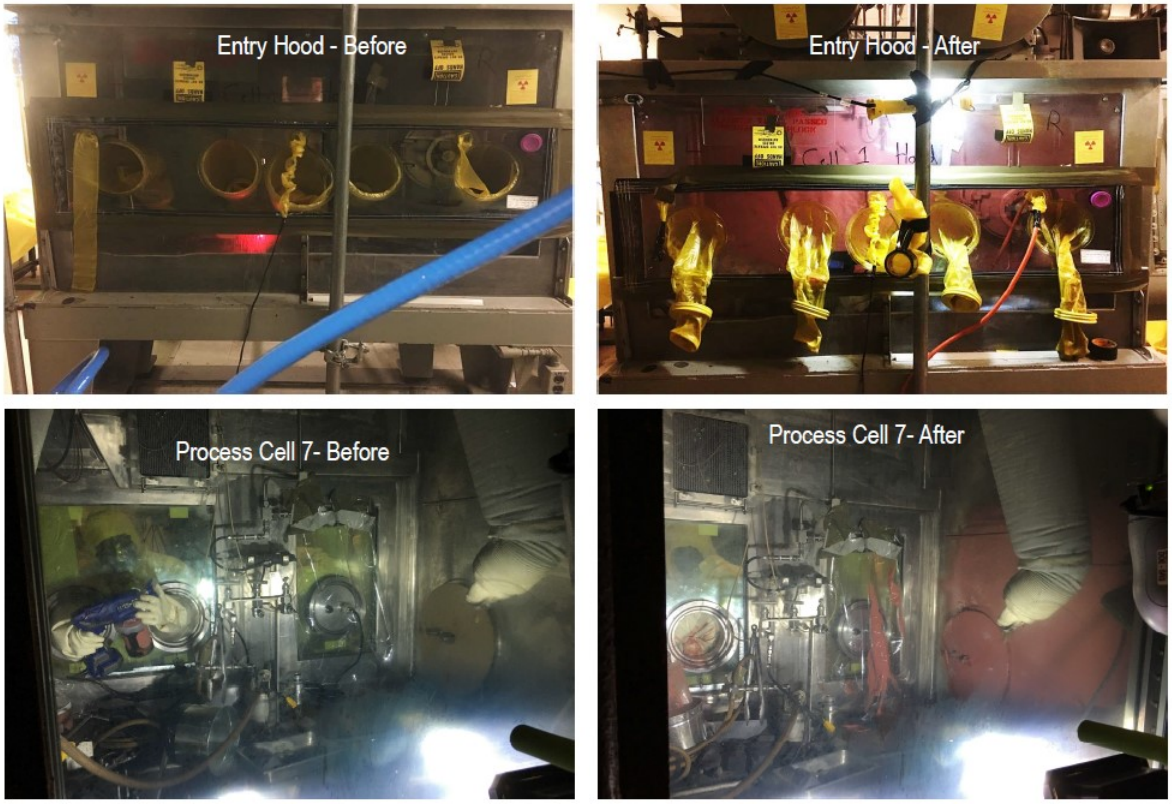


Figure 7-1. Before and After photos of fixative application of Entry Hood and Process Cell 7.

7.1 Preparation of Cells

Prior to beginning work in these areas, masking had to be installed to protect certain assets that facility personnel wanted to retain access to. This included a square door in the back of the Entry Hood (masked with plastic), the electrical passthrough and associated window (masked with plastic), and the circular passthrough leading from Process Cell 7 to 8 (masked with cardboard). Also, to maintain confinement of any material that may be stirred up during activities, a flex panel glove wall was installed on the Entry Hood opening and can be seen in Figure 7-1 (Entry Hood-Before Photo). Debris was also removed from both the Entry Hood and Cell 7 floor to clear a working space.

7.1.1 Prepare Tools

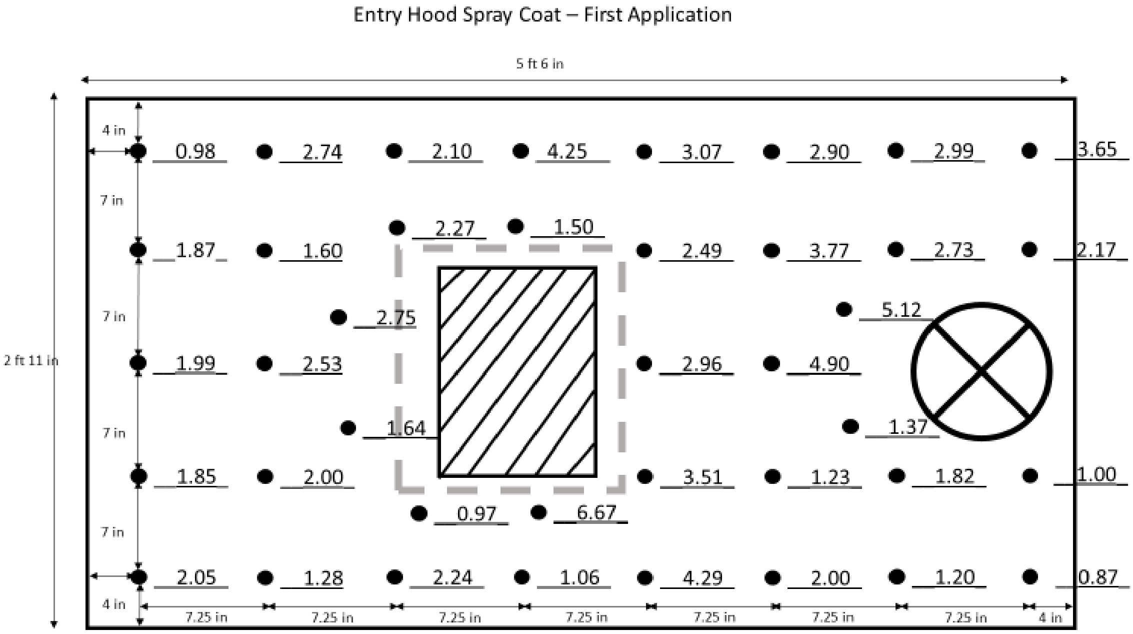
In addition to cell preparation, tools were prepared outside the line including assembly of equipment (sprayer and spreader) used in the application. Once tools were prepared, they were bagged into the cell. Prepared canisters of Product H for both pouring and spraying were bagged in bulk into Cell 7 to ensure appropriate amounts of material could be quickly accessed, along with canisters of water to purge the nozzle of the sprayer after application was completed for the day and an associated bag of desiccant to spray the water into. The sprayer cartridge was attached to the sprayer once both pieces were bagged into the cell.

7.1.2 Application

Initial efforts focused on the Entry Hood due to ease of access. The back wall was first coated completely through three applications to above the 1/8 in (0.318 cm) thickness over the course of 3 weeks with a week drying time between applications. This week drying time was found to be necessary due to inherent facility conditions such as minimal air flow in the Entry Hood that rendered the environment nearly stagnant and a downed chiller that occurred twice that resulted in extreme moisture content in the air. At times of chiller outage, standing water was observed within the facility both times, precluding personnel entry. Spray time for one bottle with one person swapping between gloves was about 4 minutes. Approximately half of the bottle was sprayed in the first position. The remainder of the bottle was sprayed from the second position. The cartridge was changed in the first position, and the spray application was repeated. Approximately 1.5 gallons was sprayed over 2 sessions and followed with a third session where 1 gallon was sprayed on targeted areas to reach the requisite thickness. The decision to perform this application in two (2) sessions was made by the operators who, having participated in the mock-up dry run described in 6.4, noted precursor signs that would have likely led to sagging/sloughing.

Once the back-wall coating was complete, the floor was coated via a pour and spread method wherein the total volume necessary to coat the floor to 1/8 in (0.318 cm) was poured and pushed along the floor with a spreading tool. Some additional material was also poured at the same time to compensate for losses to container walls and incomplete pouring from the host container. Approximately 1 gallon was poured across the floor by one person. The spreading tool was then introduced to the entry hood and used to push the material along the floor surface until visually level. This process was performed in approximately 10 minutes. After one application, all measured points were found to be of the correct thickness. The decision to perform floor application after wall application was made to allow for a working space for tooling in the confined space. It should also be noted that a continuous air monitor was directly placed to the left of the entry hood near the lower left corner of the flex wall, the same position that all tooling and cartridges were introduced through. At no time during application activities did the continuous air monitor alarm, confirming appropriate containment was achieved by the flex wall during these activities.

Once all applications were complete and full cure was achieved, measurements were taken at various points across the coated surface and are shown in Figure 7-2 and Figure 7-3 below. One benefit of taking measurements and recording them in a visual grid was that it allowed operators to visualize the areas that were not sufficiently thick and apply more material in a targeted manner. It also showed the operators areas where they sprayed from one glove position trying to maximize the amount of material applied that was subsequently resprayed by another operator in the second glove position.



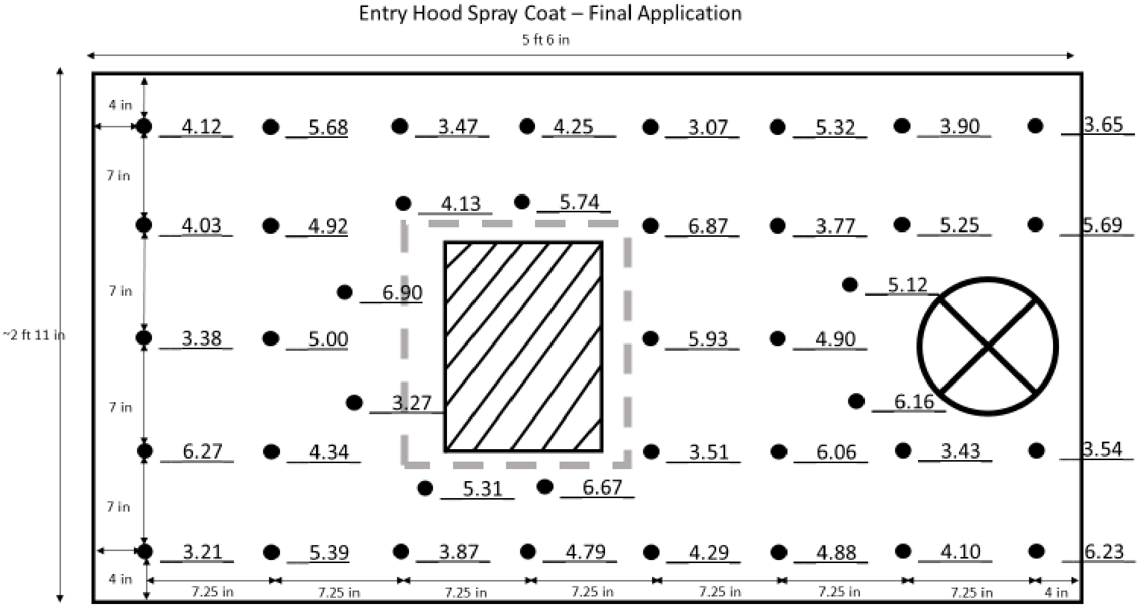


Figure 7-2. Measurements of Spray Application Thickness on Entry Hood Back Wall after Initial Application (top) and Final Application (Bottom). All Measurements are Reported in mm.

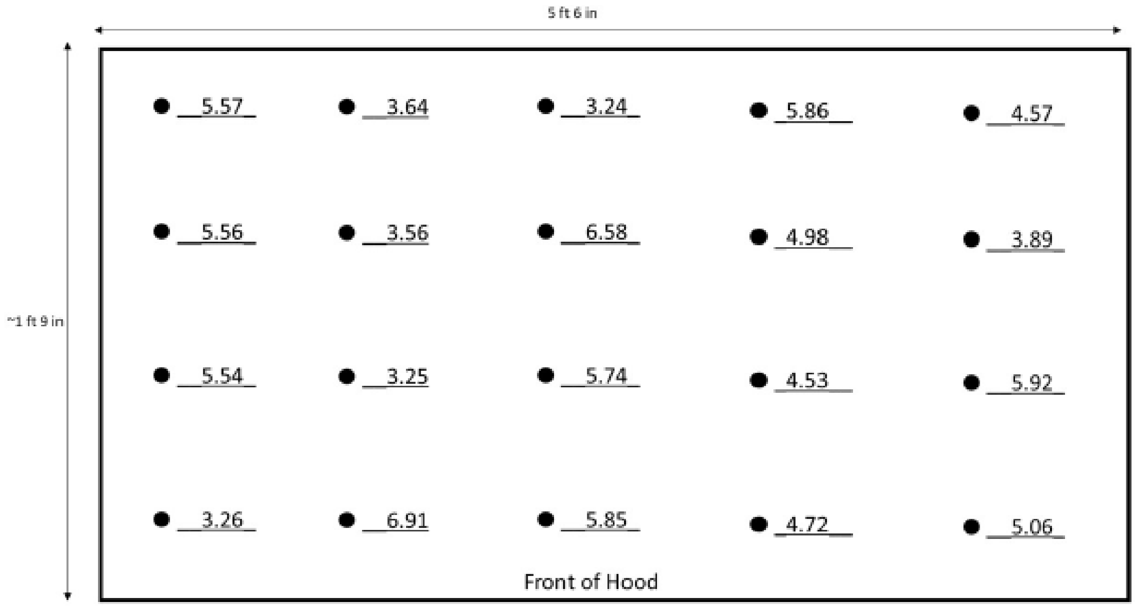


Figure 7-3. Measurements of Pour and Spread Application Thickness on Entry Hood Floor. All Measurements are Reported in mm.

7.2 Process Cell 7

During drying times for material applied in the Entry Hood, activities were performed in parallel in Process Cell 7 when the building was not inaccessible due to chiller outage. The vertical portion of the cell was found to meet thickness specification after 2 coatings. The coating of the floor was approached with the same method as the Entry Hood floor. While activities in the cold mockup were performed without any incident, significant impedance was found within the hot cell that was not considered in either of the mocked scenarios. Of particular note is the metal post with valves and hoses coming off of it in front of the glove coupled with the plastic covering on the smaller window (Figure 7-4). This post limited range of motion of the operator who was spraying, leading to moderate difficulty in ensuring complete coverage of the area to be sprayed and induced some spray onto the plastic shielding the electrical connections. While the operation was still able to be successfully completed, these were operational difficulties that were not considered until the operation was going live.

Figure 7-4. Comparison of spraying activities performed in the cold cell mockup (left) and inside Process Cell 7 (right).

8.0 Post-Application Evaluation

Long-term thickness testing was carried out on the Product H application in Building 235-F in December 2020. After initial application of Product H in the Entry Hood in 2018, the coating was left mostly undisturbed after the post-application cleanup. This timeframe was not anticipated, but necessary due to lack of funding to complete final observational data collection. During the 2 years the Product H remained in place, several modifications were made to the Entry Hood as part of the decommissioning process of Building 235-F. The first modification was the removal of the glove port wall which exposed the Entry Hood to atmosphere in 2018. The second modification was the removal of the plastic covering of the square access door to the Entry Hood, which was carried out in 2019. Additionally, the Product H application equipment stored in the floor of the Entry Hood was removed after roughly a month.

Once the application of Product H had been completed in Process Cell 7, the hot cell also underwent a continued clean out to remove equipment. At the time SRNL was able to reengage with 235-F personnel for thickness measurements, Cell 7 had reached complete closure, disallowing operators from performing work within. This made Process Cell 7 unavailable for direct thickness testing between the application in 2018 and the final thickness tests in December 2020. While thickness measurements could not be made, the status of the coating was recorded visually from the manipulator side of the hot cell.

8.1 Entry Hood Thickness Testing

Visual inspection of the Product H application in the Entry Hood showed running on the back wall and a pooling or thickening of the coating on the floor of the Entry Hood. Manufacturer provided values for Product H curing was 48 hours, however the running on the side wall application suggests curing/settling to a finalized thickness could take longer than was quoted, especially in times of excess humidity and stagnant air flow, though this behavior was not observed in testing performed on Product H similar to that described in Section 4.4.1.[29] The post-application cleanup in the Entry Hood caused significant thickness changes in coating. Figure 8-1 shows the access door after removal of the plastic covering. It can be seen that removal of the plastic covering also removed a significant amount of the intumescent coating due to the cohesion of the Product H coating, however functionality of the door was maintained by this protection and subsequent removal. Despite removal of the plastic covering, the Product H application maintained its general cohesion, and the thickness of the coating that was not stripped off by the removal of the plastic access door covering remained relatively consistent, as seen in Figure 8-2.

Figure 8-1. Photo taken of Entry Hood side door during December 2020 assessment of Product H.

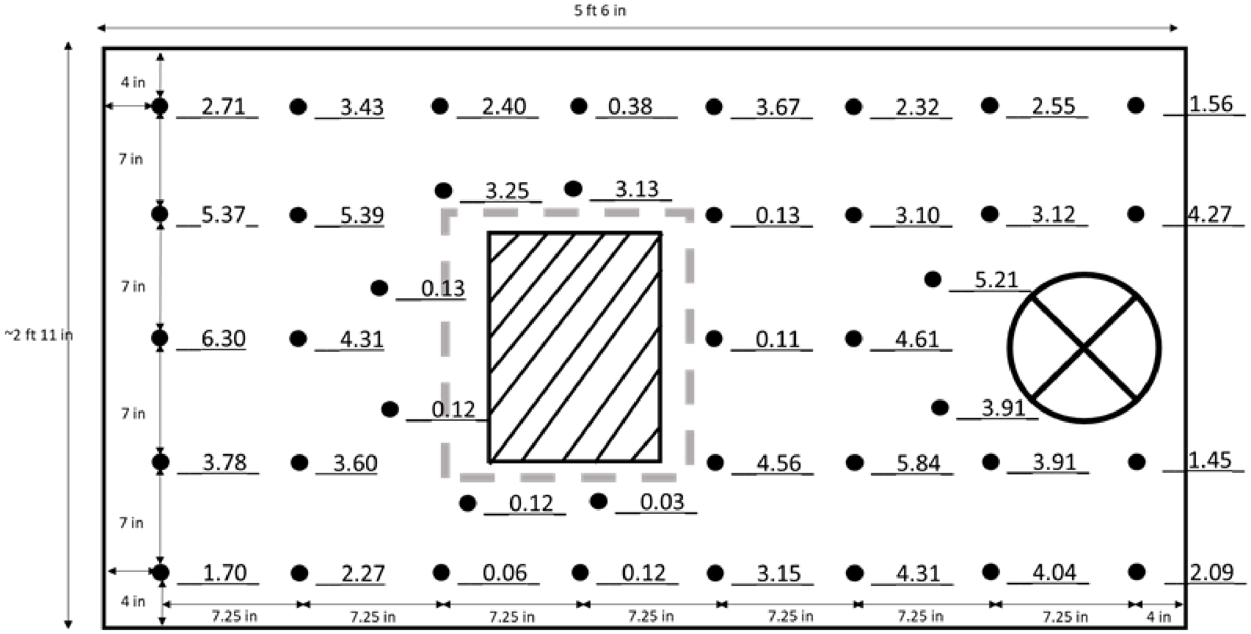
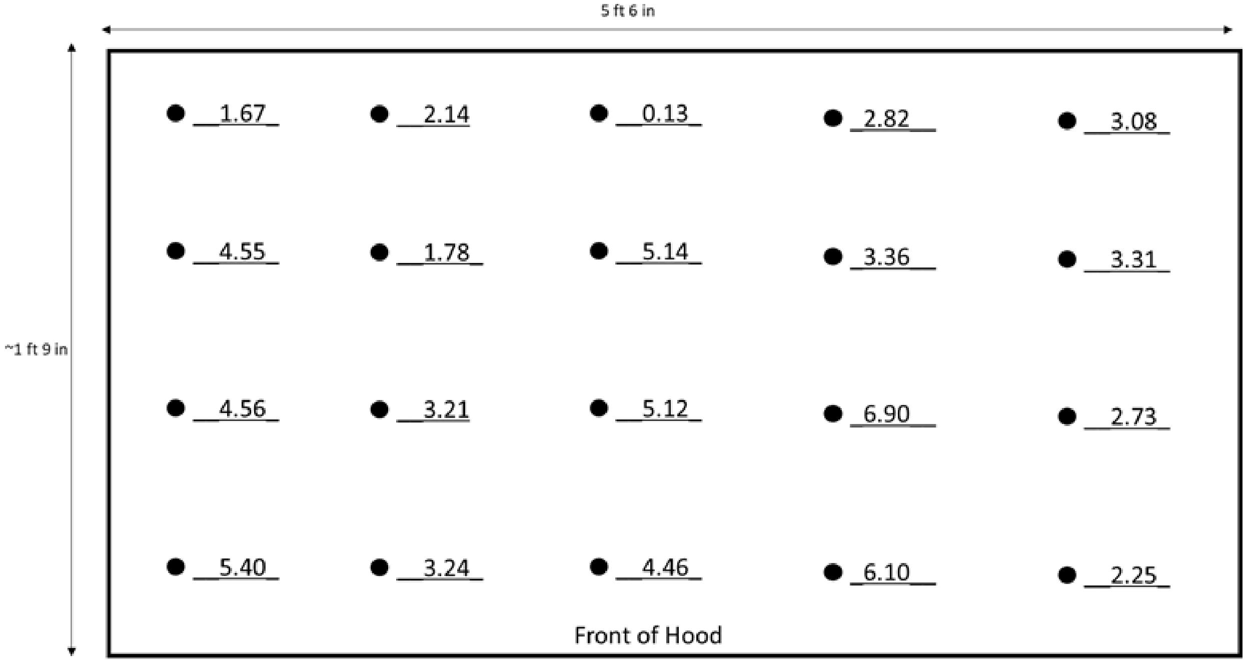


Figure 8-2. Thickness measurements made during December 2020 of the Entry Hood back wall. All measurements in mm.

The floor of the Entry Hood under the cohesive layer on the wall was mostly smooth and intact, although several indentations/divots were noted where the application equipment was kept on top of the coating after application, suggesting that the curing process continued for the month or so after the application equipment had been stored (Figure 8-3). To avoid indentations in future applications of Product H on horizontal surfaces, it is recommended to keep the surface clear for at least two months to ensure a full settling has taken place per the FIU control data set. The Entry Hood floor also had cracking noted in the coating under the round door on the right side of the hood, not shown in Figure 8-4. The indentations caused by the application equipment resulted in a thinning of some measured points in the hood as shown in Figure 8-5, with some other areas gaining thickness though not in a 1:1 ratio of losses to gains between the initial thickness measurements and the final thickness measurements suggesting compression of the coating or a reduction in thickness due to loss of water.

Figure 8-3: Visual inspection of the Entry Hood showing access door, coated wall, recovered square door, and floor of hood (partial).

Figure 8-4. Visual inspection of the Entry Hood showing access door and floor of hood and a close-up of the indentations left by the application equipment (center) and cracking under round door (right).



1.67	2.14	0.13	2.82	3.08
4.55	1.78	5.14	3.36	3.31
4.56	3.21	5.12	6.90	2.73
5.40	3.24	4.46	6.10	2.25

Figure 8-5. Thickness measurements for the Entry Hood floor taken during the December 2020 post-application assessment of the Product H coating.

The cracking found on the Entry Hood floor occurred exclusively under the round door of the hood (Figure 8-6). Inspection of the cracks revealed a white powdery residue that was determined to be the absorbent used during the regular cleaning of the application equipment. The spray applicator was cleaned after each application to reduce clogging and maintain a consistent spray. Product H also uses water as the primary solvent and maintains a level of interstitial water bound in the polymer to enhance its intumescence. The application and cleaning procedure were done while the glove port wall was still attached to the entry hood and required 2 workers to access the applicator while triple gloved. The loss of dexterity between the team that cleaned the applicator resulted in the spilling of the absorbent powder used for absorbing the cleaning water. The spilling of absorbent is localized due to the left operator holding the sprayer and right operator holding the absorbent bag during all cleaning routines. Due to the presence of the absorbent, the water was pulled from the polymer during the extended curing process and induced cracking in the final cured material.

Figure 8-6. Cracks and absorbent found directly under the round door space in the Entry Hood.

8.2 Process Cell 7 Visual Inspection

As stated previously, Process Cell 7 was unable to have thickness measurements taken due to the final closure of the cell in 2020. Visual inspection of the floor of the cell showed a significant amount of cracking similar to what was seen in the Entry Hood under the round door, and this was confirmed to be the area where a bag of absorbent had been dropped during a nozzle cleaning procedure (Figure 8-7). This cracking was only seen under the dual glove port in Process Cell 7, which were the gloves used during the cleaning processes. Operators noted it was impossible to remove the absorbent after the spill due to the then tacky nature of Product H that had recently been applied. The single glove port on the opposite end of the cell and the wall near it (Figure 8-8) showed no running, cracking, or depressions. This area of the cell was kept completely clear of all activities after the application of the Product H as all further activities were carried out in the dual glove port. This supports the theory that, absent absorbent or equipment placement on top after application, this material can provide a continuous fixating layer that maintains the manufacturer specified thickness, and thus retaining all fire standard certifications.

Figure 8-7. The dual glove port side of Process Cell 7 sustained cracking in the coating due to the absorbent left on the surface of Product H.

Figure 8-8. The single glove port side of the cell shows no running or cracking and is free of indentations.

8.3 Radiological Survey of Entry Hood

Initial surveys of the contamination in the area were taken in October 2018 and established baseline contamination levels prior to the application of Product H to the Entry Hood and Process Cell 7. The survey was carried out by measuring large area dose rates in front of the Hood and the surrounding area and the reporting was done using the Visual Survey Data System (VSDS) shown in Figure 8-9. The general area dose rate located near the hood was non-distinguishable above background radiation. A survey of the hood found a maximum of 40,000 dpm alpha via direct probe survey (Figure 8-9 point 1 diamond), and 8000 dpm alpha per 100 cm^2 for the large area survey using smears and wipes (Figure 8-9 point 8 square and circle). The work area was surveyed for transferable contamination and found 2000 dpm alpha per 100 cm^2. Of note, the survey prior to application was taken after debris had been removed, and no gross decontamination was performed on the Entry Hood prior to Product H application.

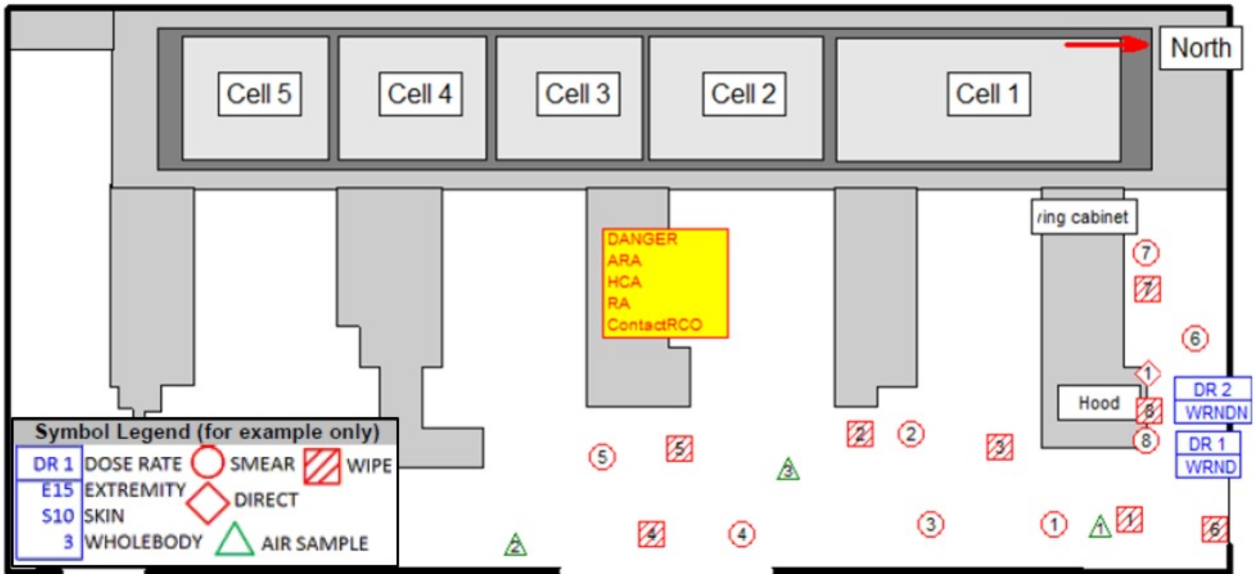


Figure 8-9. VSDS map for the initial October 2018 contamination survey.[30]

During the December 2020 post-application thickness testing, a radiological survey was carried out to assess the contamination levels present in and around the Entry Hood and displayed on a map using the VSDS system (Figure 8-10). Measurement of the same areas (noted as point 16 diamond, circle and square) found a maximum of 1000 dpm alpha per 1000 cm^2. The lower readings are indicative of the effectiveness of Product H as a coating as no decontamination or activities have occurred in the Entry Hood since cleanup of initial activities concluded and a reduction by half of the contamination measured was noted after two years.

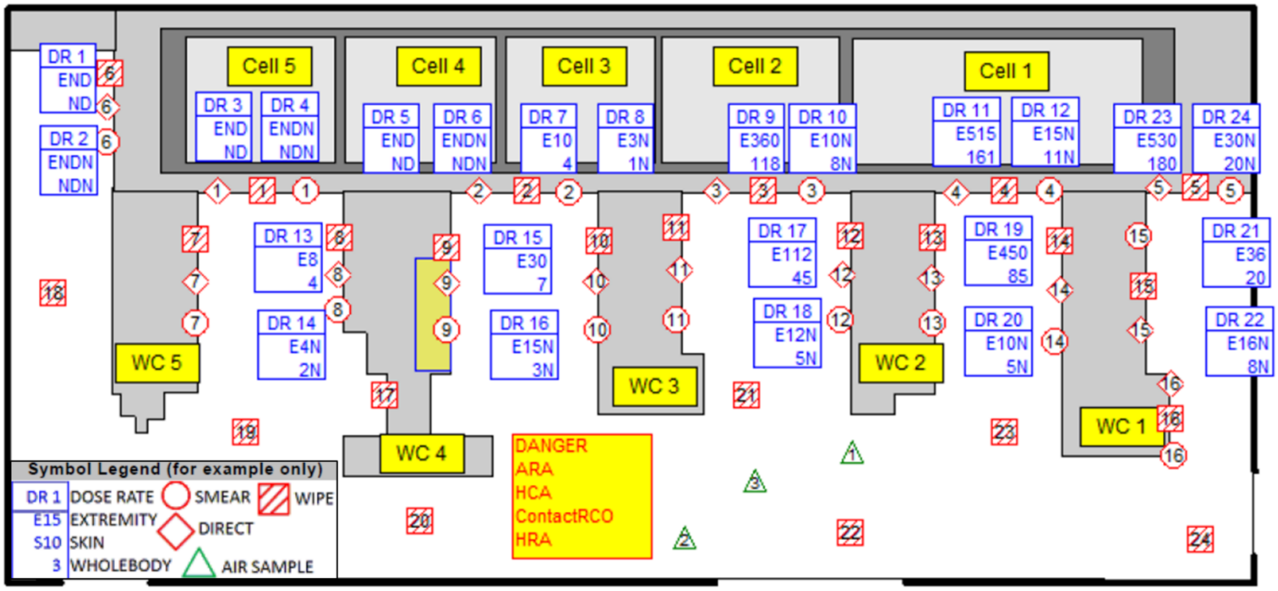


Figure 8-10. VSDS map for final contamination survey.[31]

9.0 Lessons Learned

While the results of this research activity demonstrated that the commercially available Product H intumescent coating has excellent potential for use in radiologically contaminated environments to reduce the risk of release in the event of seismic or fire activity, there are also valuable pieces of information that should be captured about both technical and institutional challenges from this project. The lessons learned from each stage of the process improved the outcomes of the next stage. The down selection allowed the FIU team to focus on application methods for only one material, FIU cold demo improved the SRS cold demo by preventing unnecessary experimentation in the mockup, and the SRS cold demo helped ensure that the hot test inside 235-F was able to be performed safely and effectively.

As an example, the FIU test results showed that the Graco Ultra brand sprayer performed better than traditional painting due to time associated with coating: 1 qt (0.95 L) of IC could be sprayed over the course of about a minute and a half with reloading time of cartridges averaging about two minutes whereas a roller method required significantly more physical effort and time to put equivalent material on the coated surface. Maintaining control of the spraying pattern was also found to be relatively easy with minimal overspray in the training environments. For horizontal coating, the pour and spread method was found to be better than spraying or rolling onto the surface. Due to the weight of the sprayer and the angles involved for the floor, the sprayer was disqualified as a means for coating. Traditional paint rolling also exhibited the same problems as with vertical surfaces: the amount of time and effort it took to coat the floor versus the pouring method proved to be excessive.[26]

These lessons applied to the SRS cold demonstration allowed operators to focus on learning the necessary methods for achieving the best application and determine 1) whether the coating method was able to achieve the requisite 1/8 in (0.318 cm) coating from the gloveports, 2) how well the associated tooling (sprayer, cartridges, spreader, thickness gauge) could be manipulated from the gloveports, and 3) identifying any additional obstacles that came from working within the enclosed space through gloves.[26] The lessons from the hot demo are captured below.

9.1 Technical Lessons Learned

9.1.1 Tools

Of note is the masking covering the electrical passthrough in Process Cell 7. Due to the constraints with the metal stand that was not on facility drawings, and thus not planned for, covering the electrical pass through got more material than anticipated sprayed on it. This in turn caused the plastic coating to fall due to the increased weight of the IC layered on the plastic. It was suggested by the operations personnel from 235-F that the masking needed to be rethought and better designed to both a) be easier applied to the protected area and b) better withstand weight of any material that is sprayed onto it.[26] An alternative to consider could be a suction cup or hook and loop solution for better positioning and to protect any structures that still need to be accessed that will better resist sagging due to the weight of the coating.

Because of the confined spaces inside the hot cell, a shorter spreader handle would also improve the ability to quickly, evenly, and easily spread the coating on the floor of the cell. A smaller spreader would also make it easier to address finer details around protrusions. From the hot test, it was noted that the sprayer should be fully assembled except for the cartridge before passing into the cell due to space constraints.

While this was performed prior to bagging into the hot cell, it was noted when initiating spraying that the nozzle was rotated 90° from what had been practiced, requiring a time out, additional bagging in of Allen wrenches, and modification of the nozzle orientation while quadruple gloved (2 pair on operator, cell glove, and over leather glove to prevent puncture). This was extremely difficult for the operators and took a significant amount of time.

9.1.2 Methods

Spray distance has an impact on application quality. FIU determined an optimal spray distance of 3 ft (36 in or 91.5 cm). From the coating of the entry hood, it was noted that the standoff distance from the flex wall to the back wall was closer than the suggested distance of 3 ft (36 in or 91.5 cm) for the Graco sprayer. This had some minimal impact on the spraying operations in that the amount of arm movement necessary to ensure a full coating was exaggerated over what could be expected from a farther standoff. Conversely, in Process Cell 7, the standoff was greater than the suggested distance of 3 ft (36 in or 91.5 cm), and lead to more than anticipated overspray of the designated spraying area. The ease and timeliness of application from the sprayer made up for these short comings, and operators were able to easily adjust to ensure full coating resulted.

Operators applying Product H in the Entry Hood had difficulty in determining a mid-line on the back of the wall due to the two-operator application method used, and with the complex geometry associated with the plastic covering and various door protrusions, portions were either over- or under-sprayed during initial application. Practicing a more systematic application method could have helped account for this prior to beginning.

Absorbent should be limited, and while water must be run through the sprayer to maintain functionality, spilling of the absorbent can significantly impact the final state of the cured fixative. Similar consideration should be given to ensuring there is a place for equipment rather than on top of the applied material to prevent significant depressions in the final cured coating.

A way to gauge material thickness before curing could reduce the number of applications and material required. Personnel used "gut-feeling" to assess material thickness in the hot demo. Multiple applications were needed on the vertical surfaces to achieve requisite thickness. Overpour (additional material added to compensate for loss to container walls) was used on the horizontal surface to achieve the required thickness in a single application due to greater than anticipated residue in the containers.

It was noted that curing was slower than expected due to inhibited air flow. Initial cure time in mockup environments was ~24 hours, however in facility hot demo, all applications required at least 48 hours to cure. It could also be more beneficial to coat the most contaminated surface (typically the "floor" and other horizontal surfaces) first to minimize disturbance of residual contamination.

9.1.3 Safety

Product H was compatible with the hot cell/flex wall gloves for this process. Residual material on the gloves was not an issue and did not induce any embrittlement/punctures. The glove did not stick to itself during application and remained flexible and uncompromised throughout the process over multiple weeks. This became important because Product H was applied in a thin layer to both hot cell gloves due to a mishandled container despite operators making every effort to maintain clean gloves.

Value of the flex wall in containing contamination within the Entry Hood cannot be overstated. No contamination was found on any of the workers or the surrounding work area throughout the 3 weeks of activity performed in this location. Likewise, no air monitoring alarms sounded from this work which historically had not been the case with this location prior to the entry hood being installed; this entry hood was a known source of particulate that induced nearby air monitoring alarms without any workers even present.

Having a spotter on the other side of the cell aided completion due to lack of visibility of the worker and limited lighting. Considerations for movement in tight spaces should also be taken into consideration in job planning. The knowledge and hands-on experience gained through the process of the cold demo was critical to the success of the personnel who conducted the hot demo, particularly in the absence of standardized methods and prior experience with this material for D&D.

Walking down the operational space is also important to verify existing conditions. As stated earlier, the metal rod with valves and tubing was on no facility drawings and was an unanticipated obstacle to material application.

9.2 Systemic Lessons Learned

A lack of international protocols can impede gaining support and buy-in for "new" technologies or expansion of technology applications. Examples of lacking protocols at the time of the hot test were: confirmation of curing of an intumescent coating, adhesion testing for fixative technology, and verification of integrity and functionality when exposed to seismic/impact stressors. This identified gap prompted FIU ARC and SRNL collaborators to develop "best fit" protocols for initial testing purposes. However, given the importance of this data to support safety basis calculations, the requirement for a concerted R&D effort to develop formal testing protocols and experimental designs that can be promulgated by international standards organizations is evident. It should be noted that some attempted "best fit" protocols resulted in technical passes as stated in the standard, however, did not meet other requirements associated at the facility level, further emphasizing the importance of D&D specific standards associated with relevant technologies such as fixatives.

The importance of fully leveraging standards-based performance specifications and testing protocols was made readily apparent and emphasized throughout the development process by site personnel. It significantly reduces risk for the end-user through uniformity, standardization, and quality assurance in technology test and evaluation. This facilitates the given technology's application across the DOE EM Complex with little to no recertification required. Though an acceptable approach in some instances, the results do not carry the same accepted credibility as those produced from internationally promulgated protocols and make it increasingly difficult to readily deploy the technology and/or apply the method across the DOE EM Complex. Continued engagement with international standards organizations is required and developing uniform testing protocols in key areas associated with fixatives such as adhesion testing, determining fixating capacity under thermal and seismic stressors, resuspension factors (to include airborne release fractions and respirable fractions), etc., is needed.

10.0 Conclusions and Next Steps

During the execution of this test plan, several areas were identified that warrant future research, development, testing, and evaluation. All the topics below have been discussed in detail with stakeholders at all levels, and there is consensus that investment in this direction would represent the next logical step in addressing several of the gaps and shortfalls identified in this document.

10.1 Targeted Research to Provide Empirical Data to Update DOE-HDBK-3010

As identified by SRNL, FIU ARC, and SRS, the current version of DOE-HDBK-3010 has not been revised since it was initially issued in 1994 and does not account for fixative technologies and their potential impacts on the source term formula. Safety basis calculations support the safety considerations necessary for legacy nuclear facilities as they transition from active use, through limited operations and standby modes, until final disposition is achieved. Many of the calculations involve estimating doses to a collocated worker at 100 meters from the source term and at the site boundary. These dose values are governed by the values presented in DOE-HDBK-3010 in the form of airborne release of radioactive material resulting from penetration of the facility per seismic activity, full facility fires, and/or explosions. For radioactive material in the form of stored metals, liquids, or powders, no standardized test methods or empirical data exist for quantifying key aspects of materials, such as fixatives and intumescent coatings, and their associated impact on ARF/RFs should a change of state occur such as fixation of a powder in a polymer layer. DOE-HDBK-3010 does not take into account a change of state as would be proposed through this work; the powdered residual radioactive material would be immobilized in a polymer platform, and thus should be considered a solid rather than a powder. This change of state would significantly reduce the ARF/RF values, leading to a more accurate reduced MAR through this mitigating method. While the above work outlined in this document took a COTS technology from a technology readiness level 1 to 7 for this application, more empirical data is still required to better define and benchmark how these materials work in a finite manner.

To this end, FIU ARC, in close collaboration with DOE EM, SRNL, and other stakeholders, has initiated a research activity under its current Cooperative Agreement centered on ascertaining the positive impacts fixative technologies in general, and fire retardant fixatives in particular, may have on immobilizing residual contamination once encapsulated in a solid polymer material. It is hypothesized that this solid-state polymer form will significantly mitigate the potential of release under not only normal operating conditions, but contingency-based scenarios postulated in BIOs across the DOE complex as well. This change of state resulting from the application of fixative technologies should significantly reduce the ARF/RF values, leading to a more accurate reduced Source Term through this mitigating method. It will also facilitate quantification of fixative technologies designed to mitigate contingency scenarios outlined in DOE Complex-wide BIOs and lay the groundwork for uniform international standards and testing protocols through ASTM. Such data will allow for much more rapid integration of these materials, allowing for formal crediting of the fixative platforms and subsequently reducing the cost to maintain the facility due to a lower safety consequence in the event of facility breach.

The research activity will evaluate fixative performance under ideal conditions and when exposed to a variety of stressors (i.e. impact, environmental such as water immersion, thermal / fire, etc.) associated with safety basis analysis in response to high priority requirements identified across the DOE Complex. This research effort has the added benefit of potentially providing essential data points on the positive effects of fixative technologies on mitigating ARFs, RFs, and resuspension rates used in safety basis calculations and the Source Term Formula outlined in DOE-HDBK-3010. This, in turn, will provide the foundation for providing a uniform methodology for certifying fixative technologies.

10.2 Development of Uniform Testing Protocols & Performance Metrics for D&D Technologies

The importance of fully leveraging standards-based performance specifications and testing protocols was consistently emphasized throughout the test and evaluation process by safety basis personnel and end users. Adherence to ASTM/NIST/ISO standards significantly reduces risk for the end-user through uniformity, standardization, and quality assurance in technology tests and evaluations. In several instances, however, uniform testing protocols for critical aspects of evaluating fixative technologies were not available, requiring FIU ARC and SRNL researchers to develop "best fit" procedures. Though an acceptable approach in some instances, the results do not carry the same accepted credibility as those produced from internationally promulgated protocols and make it increasingly difficult to readily deploy the technology / apply the method across the DOE EM Complex. Continued engagement with international standards organizations is required and developing uniform testing protocols in key areas associated with fixatives such as adhesion testing, determining fixating capacity under thermal and seismic stressors, resuspension factors to include ARFs, RFs, etc., is needed. Focused research on the experimental design to address the above gaps, and successfully navigating the consensus-based standards development process of international standards organizations, would facilitate the operational deployment of new D&D technologies.

There is extensive literature by many innovation scholars supporting the general role of standards as powerful institutional mechanisms that shape technological innovation, and particularly diffusion. A full accounting of this research in other industries far exceeds the parameters of this document. However, they are overwhelmingly consistent in highlighting the crucial function consensus-based standards play in reducing risks and uncertainties by communicating information and providing appropriate legitimacy to new technology systems. In particular, by establishing coordinated acceptance of technical norms, they develop positive externalities in the form of network externalities, therefore increasing the attractiveness of the technology for the customers and leading to rapid diffusion of innovation systems.[32-33]

Figure 10-1 depicts the multiple tasks performed by standards as they transcend the three major stages of technology-based activity - R&D, production, and diffusion/market penetration.[34] Of particular note, for risk-averse industries such as the D&D of nuclear facilities and sites, their role in risk reduction, and specifically in building trust, serves as a critical enabler to mitigating information asymmetry and enhancing the given technology's diffusion into the operational environment. The four broad functions of standards are outlined in Figure 10-2, and Tassey provides a detailed explanation of how each of these interacts throughout the technology development and diffusion process. In terms of risk reduction, standards specify acceptable product or service performance along one or more dimensions such as functional levels, performance variation, service lifetime, efficiency, safety, and environmental impact.[34]

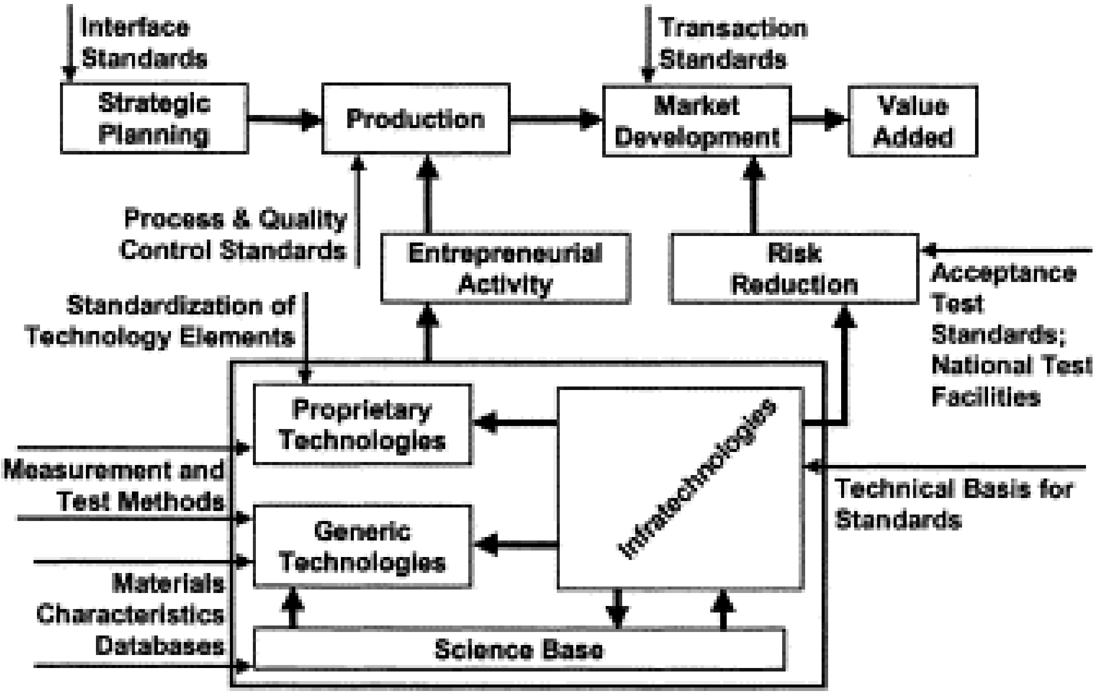


Figure 10-1. Roles of Standards in a Technology-Based Industry.[34]

	Role of Public Policy to Promote Innovation
Compatibility / Interoperability	• Initiate new standardization processes in case of lock in in old technologies in case of strong network externalities • Ensure compatible and interoperable solutions , e.g. by policy initiatives like the European Interoperability Framework • Promote network externalities by restricting IPR in standards
Minimum Quality/ Safety	• Involve all stakeholders in open, transparent and consensus-based standardization processes • Initiate the development of performance instead of design standards • Reference standards asking for high quality in public procurement
Variety Reduction	• Initiate standardization processes including all relevant stakeholders, incl. public procurers, to develop – if possible technology-neutral – standards in order to promote critical mass in emerging technologies and industries in due time without selecting prematurely specific technologies • Reference standards in public procurement processes to promote the development of critical masses • Ensure that standards including IPR can be implemented by all interested companies in order to avoid market concentration
Information	• Promote the transfer of research results into standardization processes and standards supported by public support programs • Promote the diffusion of the content of standards

Figure 10-2. Four Functions of Standards and Role in Promoting Technology.[34]

There are several federal directives that recognize the essential role of standards. For example, Section 12(d) of the National Technology Transfer and Advancement Act (Public Law 104-113), directs federal departments to achieve a greater reliance on voluntary consensus standards, and this mantra is also required by the Office of Management and Budget's Circular A-119,"Federal Participation in the Development and Use of Voluntary Consensus Standards and in Conformity Assessment Activities".[35-36]

Given that standards-based testing and evaluation is a critical enabler to the successful transition and deployment of D&D technologies from the lab to the end user, FIU, SRNL and DOE EM will continue to actively participate in ASTM International's E10 and E10.03 Committees to develop and promulgate uniform performance metrics and testing protocols for D&D technologies, with a particular emphasis on fixatives.

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Appendix A: Work Package 01648596-01

Asset Suite Information

Date Created	10/01/2018
Planner Name	Susie Huff
Planner Telephone	2-3534
Equipment Name	MISCELLANEOUS GS PUFF COMPONENTS
Facility	V35
CLI Number	FP - 235000 - PUFF – GB - 7
Functional Class	SS

1.0 Scope of Work:

The work scope is to apply Product H to select surfaces of Cell 7 and the Entry Hood (exterior component of the Cell 1 Cold Press Glovebox) in accordance with SRNL-TR-2018-00074. In addition, the work scope is to temporarily place and operate lab-scale electrostatic precipitators (ACE 2.0) in the same spaces also per SRNL-TR-2018-00074.

2.0 Precautions and Limitations:

2.1. Safety System Impact Statement: There are no safety system impacts identified with this work activity.

2.2. Employees should be cognizant of and self-monitor for signs/symptoms of Heat Stress or Cold Stress.

2.3. Control of combustible material will be handled using procedure, 221-F-51105, “F-Area Complex Controls and Limits of Combustibles.” (Note that Product H is not considered a combustible material.)

2.4. When work is being performed in Building 235-F, the PIC/Work Group Supervisor shall ensure at the end of each day’s activities that all transient combustibles are removed from the building, stored in a metal container, or approved in accordance with FRM-235-F-215, Building 235-F Designated Transient Combustible Posting.

2.5. Personnel should not attempt to lift more than they are capable of lifting. If equipment is hard or awkward to handle, mechanical means should be used and/or additional personnel should perform the task.

2.6. Proper lifting techniques should be used. Loads should be lifted with the legs instead of the back and twisting or bending at the waist should be avoided.

3.0 Prerequisites: Initials

3.1. WGS/POC: **CONDUCT** the pre-job briefing with affected personnel prior to starting work, **AND**

ENSURE the following areas have been addressed in the Pre-Job Briefing:

- AHA Hazards and Controls
- Sign onto appropriate RWP
- Tools and materials are staged at work location
- Work support personnel (IH, RadCon, Operations, etc.) have been contacted
- Prefabrication work is complete
- Cut and puncture hazards including a review of 235-F-3644, Puncture/Laceration Wound Hazard Management Program ______

3.2. WGS/RR Worker: **REVIEW AND UNDERSTAND** instructions for operation of the Graco Ultra Sprayer and the Defelsko PosiTector-6000 FNTS dry film gauge. ______

3.3. WGS/RR Worker: **RECEIVE** briefing from SNRL on the proper operation of the ACE 2.0. ______

3.4. WGS/RR Worker: **NOTIFY** the SOM of the work to be performed AND **ENSURE** that Work Release authorization signatures have been obtained on the Work Release Form. _________

NOTE: The following three (3) steps (3.5, 3.6 and 3.7) may be worked in any order.

3.5. WGS/POC: **ENSURE** gloves has been installed in the lower, middle and southern gloveports of Cell 7 and in the Cell 6 gloveports in accordance with 235-F-3643, PuFF Facility Glovebox/Cell Glove/Sphincter Replacement and Blind Cartridge or Electrical Pass Through Cartridge Assembly Installation. ______

3.6. WGS/POC: **ENSURE** Electrical Pass Through has been installed in the most northern lower gloveport of Cell 7 in accordance with 235-F-3643, PuFF Facility Glovebox/Cell Glove/Sphincter Replacement and Blind Cartridge or Electrical Pass Through Cartridge Assembly Installation. ______

3.7. WGS/POC: ENSURE covers (masking) have been installed over the shield window at the Northeast corner of the Cell 7 and over the Transfer Lock in the north wall of Cell 7. ______

4.0 Task Performance:

4.1. WGS/POC: **REQUEST** RPD to perform transferable contamination / dose rate surveys of work areas before performing any of the following:

- Opening any electrical box
- Opening any conduit
- Opening any closed panels
- Before starting any work above the 7 foot line (Overhead Contamination Area) within Radiological Buffer Areas (RBA)
- Any activity that can disturb painted surfaces located within RBA's

APPLY PRODUCT H TO CELL 7 AND DEMONSTRATE ACE 2.0 IN CELL 7

NOTE: Product H will be applied to the western floor (area approximately 2' by 6') and lower northern wall (area approximately 3' by 3') of Cell 7. These surfaces should be free of loose components and loose materials prior to Product H application.

NOTE: There is a shallow dam (approximately 1/2" high) on the floor of Cell 7 that runs north-south. This dam is the boundary between the western and eastern floors of Cell 7.

! WARNING !

Dilute nitric acid may be encountered in Cell 7, especially during the movement or removal of interferences. This chemical is not compatible with vacuum grease, scrubs in a bucket or Isoclean. Therefore, it must be absorbed and removed from the cells before continuing with this work instruction.

4.2. WGS/RR Worker: In Cell 7, **INITIATE** baseline operation of the ACE 2.0 by performing the following actions:

42.1. **CONNECT** power to ACE 2.0. __________

42.2. **POSITION** ACE 2.0 on eastern floor of Cell 7. __________

42.3. On Attachment A, **RECORD** date and time when ACE 2.0 is started. __________

NOTE: When removing/installing plates from/to the ACE 2.0, minimize cross-contamination of the plates by using fresh tongs and avoiding contact with gloves.

4.3. WGS/RR Worker/RPD: After approximately 24 hours, **STOP** operation of the ACE 2.0 by performing the following actions:

43.1. **DISCONNECT** power to ACE 2.0.

43.2. On Attachment A, **RECORD** date and time when ACE 2.0 is stopped. __________

43.3. **REMOVE** collection plates from ACE 2.0. __________

43.4. **INSTALL** fresh collection plates in the ACE 2.0. __________

43.5. **REMOVE** collection plates from West Line cells. __________

43.6. **SURVEY** both sides of each plate with Bicron Instrument **AND RECORD** results on Attachment A. __________

4.4. WGS/RR Worker: **MOVE** or **REMOVE** loose components/materials in Cell 7 that interfere with the application of Product H. ______

4.5. WGS/RR Worker: **IF** found during movement or removal of interferences, **THEN ABSORB, CLEANUP, AND REMOVE** free liquids from West Line cells. ______

4.6. WGS/RR Worker: **SWEEP/WIPE** the western floor of Cell 7 to remove dust, dirt and/or oil if present. ______

4.7. WGS/RR Worker: **REQUEST** SRNL Principal Investigator to inspect Cell 7, **AND ENSURE** surfaces are ready for application of Product H. ______

NOTE: An area approximately 3' high by 3' wide is to be sprayed. This area starts at the floor and northeast corner of Cell 7.

NOTE: The sprayer reservoir will be sealed with a rubber stopper to allow it to be passed into the cells. This rubber stopper must be removed prior to connecting the reservoir to the sprayer body.

NOTE: To be effective, the cured Product H must be at least 1/8" thick. SRNL-TR-2018-00074 provides an estimate of the volume of Product H to be sprayed to achieve the desired thickness.

4.8. WGS/RR Worker: Using approved tools, **APPLY** Product H to north wall of Cell 7. ______

4.9. WGS/RR Worker: On Attachment B, **RECORD** date, time, location and quantity of Product H that was applied. ______

4.10. WGS/RR Worker: **CLEAN** sprayer by running approximately 1 pint of clean water through the sprayer and by spraying into a bag filled with an approved absorbent. ______

NOTE: The Product H has cured if it can be touched lightly with a dry, white cloth and no Product H residue shows up on the cloth.

4.11. WGS/RR Worker: After at least 24 hours, **CHECK AND**—if needed—**RECHECK** the Product H with a dry cloth and **ENSURE** it has cured. ______

4.12. WGS/RR Worker: Using dry film gauge, **MEASURE** thickness of Product H on north wall of Cell 7 at locations specified by SRNL and **RECORD** results along with date and time on Attachment B. ________

4.13. WGS/RR Worker: **IF** all measurements are NOT greater than 1/8" or are not accepted by the SRNL Principle Investigator, **THEN REPEAT** Steps 4.8 through 4.12 for areas of the north wall that did not have the desired thickness. ________

4.14. WGS/RR Worker: **REMOVE** covers (masking) over the shield window at the Northeast corner of the Cell 7 and over the Transfer Lock in the north wall of Cell 7. ________

NOTE: A floor area approximately 2' wide by 6' long is to be coated with Product H in two phases by pouring Product H onto the floor and spreading the Product H with tools such as a squeegee. The initial pour will cover the northern half of the western floor of Cell 7, while a second pour will cover the southern half of the western floor of Cell 7.

NOTE: Steps 4.15 through 4.28 may be worked concurrently with 4.13 and 4.14.

4.15. WGS/RR Worker: **MOVE** or **REMOVE** loose equipment/materials in Cell 7 that interfere with the application of Product H to the western floor of Cell 7. ______

4.16. WGS/RR Worker: **INSTALL** temporary dam (1/2" aluminum bar with two-sided tape) in the western floor of Cell 7 so as to divide the western floor into two areas. ______

NOTE: To be effective, the cured Product H must be at least 1/8" thick. SRNL-TR-2018-00074 provides an estimate of the volume of Product H to be poured to achieve the desired thickness.

4.17. WGS/RR Worker: **REQUEST** SRNL Principal Investigator to inspect Cell 7 floor, **AND ENSURE** surfaces are ready for application of Product H. ______

4.18. WGS/RR Worker: Using approved tools, **APPLY** Product H to northern half of western floor in Cell 7. ______

4.19. WGS/RR Worker: On Attachment B, **RECORD** date, time, location and quantity of Product H that was applied. ______

NOTE: The Product H has cured if it can be touched lightly with a dry, white cloth and no Product H residue shows up on the cloth.

4.20. WGS/RR Worker: After at least 24 hours, **CHECK AND**—if needed—**RECHECK** the Product H with a dry cloth and **ENSURE** it has cured. ______

4.21. WGS/RR Worker: Using dry film gauge, **MEASURE** thickness of Product H on northern half of western floor at locations specified by SRNL and **RECORD** results along with date and time on Attachment B. _________

4.22. WGS/RR Worker: **IF** all measurements are NOT greater than 1/8", **THEN REPEAT** Steps 4.18 through 4.21 for areas of the northern half that did not have the desired thickness. _________

4.23. WGS/RR Worker: **REMOVE** temporary dam in the western floor of Cell 7 that divided the western floor into two areas. ______

NOTE: To be effective, the cured Product H must be at least 1/8" thick. SRNL-TR-2018-00074 provides an estimate of the volume of Product H to be poured to achieve the desired thickness.

4.24. WGS/RR Worker: Using approved tools, **APPLY** Product H to southern half of western floor in Cell 7. ______

4.25. WGS/RR Worker: On Attachment B, **RECORD** date, time, location and quantity of Product H that was applied. ______

NOTE: The Product H has cured if it can be touched lightly with a dry, white cloth and no Product H residue shows up on the cloth.

4.26. WGS/RR Worker: After at least 24 hours, **CHECK AND**—if needed—**RECHECK** the Product H with a dry cloth and **ENSURE** it has cured. ______

4.27. WGS/RR Worker: Using dry film gauge, **MEASURE** thickness of Product H on southern half of western floor at locations specified by SRNL and **RECORD** results along with date and time on Attachment B. _________

4.28. WGS/RR Worker: **IF** all measurements are NOT greater than 1/8", **THEN REPEAT** Steps 4.24 through 4.27 for areas of the southern half that did not have the desired thickness. _________

4.29. WGS/RR Worker: In Cell 7, **INITIATE** post-application operation of the ACE 2.0 by performing the following actions:

4.29.1. **CONNECT** power to ACE 2.0. _________

4.29.2. **POSITION** ACE 2.0 on eastern floor of Cell 7. _________

4.29.3. On Attachment A, **RECORD** date and time when ACE 2.0 is started. _________

NOTE: When removing plates from the ACE 2.0, minimize cross-contamination of the plates by using fresh tongs and avoiding contact with gloves.

4.30. WGS/RR Worker/RPD: After approximately 24 hours, **STOP** operation of the ACE 2.0 by performing the following actions:

4.30.1. **DISCONNECT** power to ACE 2.0. __________

4.30.2. On Attachment A, **RECORD** date and time when ACE 2.0 is stopped. __________

4.30.3. **REMOVE** collection plates from ACE 2.0 and from West Line cells. __________

4.30.4. **SURVEY** both sides of each plate with Bicron Instrument **AND RECORD** results on Attachment A. __________

NOTE: The ACE 2.0 plates are to be temporarily stored until SRNL has approved their disposal.

4.31. WGS/RR Worker: **PERFORM** waste handling, and waste removal in accordance with Procedure 235-F-WH-030, General Decontamination and Waste Removal in the 235-F PuFF Facility. __________

NOTE: Activities in the Entry Hood can be worked before, after and/or concurrently with activities in Cell 7.

APPLY PRODUCT H TO ENTRY HOOD AND DEMONSTRATE ACE 2.0 IN ENTRY HOOD

NOTE: Product H will be applied to the floor (area approximately 2' by 5') and rear wall (area approximately 3' by 5') of the Entry Hood. These surfaces should be free of loose components and loose materials prior to Product H application.

4.32. WGS/POC: **ENSURE** containment has been installed at the Entry Hood. ______

4.33. WGS/RR Worker: In Entry Hood, **INITIATE** baseline operation of the ACE 2.0 by performing the following actions:

4.33.1. **CONNECT** power to ACE 2.0. __________

4.33.2. **POSITION** ACE 2.0 on floor of Entry Hood. __________

4.33.3. On Attachment C, **RECORD** date and time when ACE 2.0 is started. __________

NOTE: When removing plates from the ACE 2.0, minimize cross-contamination of the plates by using fresh tongs and avoiding contact with gloves.

4.34. WGS/RR Worker/RPD: After approximately 24 hours, **STOP** operation of the ACE 2.0 by performing the following actions:

4.34.1. **DISCONNECT** power to ACE 2.0. __________

4.34.2. On Attachment C, **RECORD** date and time when ACE 2.0 is stopped. __________

4.34.3. **REMOVE** collection plates from ACE 2.0. __________

4.34.4. **INSTALL** fresh collection plates in the ACE 2.0. __________

4.34.5. **PROBE** both sides of each plate with alpha probe **AND RECORD** results on Attachment C. __________

4.34.6. **REMOVE** collection plates from Entry Hood. __________

4.35. WGS/RR Worker: **MOVE** or **REMOVE** loose components/materials in Entry Hood that interfere with the application of Product H. ______

4.36. WGS/RR Worker: **SWEEP** floor of Entry Hood to remove dirt and debris. ______

4.37. WGS/RR Worker: **INSTALL** covers (masking) over the large square door in the rear wall of the Entry Hood. ______

NOTE: The sprayer reservoir will be sealed with a rubber stopper to allow it to be passed into the cells. This rubber stopper must be removed prior to connecting the reservoir to the sprayer body.

NOTE: To be effective, the cured Product H must be at least 1/8” thick. SRNL-TR-2018-00074 provides an estimate of the volume of Product H to be sprayed to achieve the desired thickness.

4.38. WGS/RR Worker: Using approved tools, **APPLY** Product H to rear wall of the Entry Hood. ______

4.39. WGS/RR Worker: On Attachment D, **RECORD** date, time, location and quantity of Product H that was applied. ______

4.40. WGS/RR Worker: **CLEAN** sprayer by running approximately 1 pint of clean water through the sprayer and by spraying into a bag filled with an approved absorbent. ______

NOTE: The Product H has cured if it can be touched lightly with a dry, white cloth and no Product H residue shows up on the cloth.

4.41. WGS/RR Worker: After at least 24 hours, **CHECK AND**—if needed—**RECHECK** the Product H with a dry cloth and **ENSURE** it has cured. ______

4.42. WGS/RR Worker: Using dry film gauge, **MEASURE** thickness of Product H on rear wall of the Entry Hood at locations specified by SRNL and **RECORD** results along with date and time on Attachment D. __________

NOTE: Steps 4.45 and 4.50 may be worked concurrently with Steps 4.43 and 4.44.

4.43. WGS/RR Worker: **IF** all measurements are NOT greater than 1/8" or are not accepted by SRNL Principal Investigator, **THEN REPEAT** Steps 4.38 through 4.42 for areas of the rear wall that did not have the desired thickness. __________

4.44. WGS/RR Worker: **REMOVE** covers (masking) over the large, square door in the rear wall of the Entry Hood. __________

4.45. WGS/RR Worker: **MOVE** or **REMOVE** loose equipment/materials in Entry Hood that interfere with the application of Product H to the floor of the Entry Hood. ______

NOTE: To be effective, the cured Product H must be at least 1/8" thick. SRNL-TR-2018-00074 provides an estimate of the volume of Product H to be poured to achieve the desired thickness.

4.46. WGS/RR Worker: Using approved tools, **APPLY** Product H to the entire floor of the Entry Hood. ______

4.47. WGS/RR Worker: On Attachment D, **RECORD** date, time, location and quantity of Product H that was applied. ______

NOTE: The Product H has cured if it can be touched lightly with a dry, white cloth and no Product H residue shows up on the cloth.

4.48. WGS/RR Worker: After at least 24 hours, **CHECK AND**—if needed—**RECHECK** the Product H with a dry cloth and **ENSURE** it has cured. ______

4.49. WGS/RR Worker: Using dry film gauge, **MEASURE** thickness of Product H on floor at locations specified by SRNL and **RECORD** results along with date and time on Attachment D. __________

4.50. WGS/RR Worker: **IF** all measurements are NOT greater than 1/8", **THEN REPEAT** Steps 4.46 through 4.49 for areas of the floor that did not have the desired thickness. __________

4.51. WGS/RR Worker: In Entry Hood, **INITIATE** post-application operation of the ACE 2.0 by performing the following actions:

4.51.1. **CONNECT** power to ACE 2.0. __________

4.51.2. **POSITION** ACE 2.0 on floor of Entry Hood. __________

4.51.3. On Attachment C, **RECORD** date and time when ACE 2.0 is started. __________

NOTE: When removing plates from the ACE 2.0, minimize cross-contamination of the plates by using fresh tongs and avoiding contact with gloves.

4.52. WGS/RR Worker/RPD: After approximately 24 hours, **STOP** operation of the ACE 2.0 by performing the following actions:

4.52.1. **DISCONNECT** power to ACE 2.0.

4.52.2. On Attachment C, **RECORD** date and time when ACE 2.0 is stopped. __________

4.52.3. **REMOVE** collection plates from ACE 2.0. __________

4.52.4. **PROBE** both sides of each plate with alpha probe and **RECORD** results on Attachment C. __________

4.52.5. **REMOVE** collection plates from Entry Hood. __________

NOTE: The ACE 2.0 plates are to be temporarily stored until SRNL has approved their disposal.

4.53. WGS/RR Worker: **PERFORM** waste handling, and waste removal in accordance with Procedure 235-F-WH-030, General Decontamination and Waste Removal in the 235-F PuFF Facility. __________

4.54. Maintenance: **NOTIFY** SOM that work is complete. __________

5.0 Post Maintenance Testing & Return to Service:

None

6.0 Post Work & Feedback:

6.1. Maintenance: **ENTER** work history data into WMS considering:

- Details of the work performed
- Special equipment and tools used
- Procedures or drawings needed
- Parts and materials installed (include make and model; add manufacturer if not listed in the detail on the Requisition)
- Post maintenance testing accomplished
- Job hazards encountered
- Problems encountered and actions taken to resolve them
- Other information that may be useful at a later date should be included in the history provided by Maintenance in M350, History Brief Panel ______

6.2. Maintenance: **SUBMIT** feedback for the planners to use to improve future performance of this task or other work packages by entering a comment of type "CO" on Panel M301, for example:

- Better order of task instructions
- Changes that will make the task instructions easier to follow
- Better method of accomplishing the task
- Tools/material needed that were not identified in the work package
- Hazards encountered that were not identified in planning ______

7.0 Attachments / References:

- Attachment A, Logsheet for Operation of ACE 2.0 in Cell 7
- Attachment B, Logsheet for Application of Product H to Cell 7
- Attachment C, Logsheet for Operation of ACE 2.0 in Entry Hood
- Attachment D, Logsheet for Application of Product H to Entry Hood
- Instructions for Graco Ultra Sprayer
- Instructions for Defelsko FNTS 6000
- References
    - SRNL-TR-2018-00074, Incombustible Fixative and ACE 2.0 Test Plan
    - SDD-2015-00002, 235-F Risk Reduction Tooling List
    - SDD-2014-0067, 235-F Risk Reduction Chemical List
    - Procedure 221-F-51105 for Control of Transient Combustibles
    - Procedure 235-F-3644, Puncture/Laceration Wound Hazard Management Program
    - Procedure 235-F-WH-030, General Decontamination and Waste Removal in the 235-F PuFF Facility

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